Monitoring Coalition Program

Field Monitoring Guidance

Version 2.1

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Program Administered and Guidance Prepared by:

North Carolina
Department of Environment Quality
Division of Water Resources
Water Sciences Section
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Monitoring Coalition Program – Description:

The Monitoring Coalition Program is a voluntary, member-led, ambient monitoring program that provides an effective and efficient means for assessing water quality in a watershed context. A monitoring coalition is a group of dischargers and stakeholders that combine resources to collectively fund and perform an instream monitoring program. By forming a coalition, members have a medium to gather more information about their watersheds, evaluate member-specific interests and collaborate on watershed specific issues.

Participating members work with DWR to develop a monitoring network that uses strategically selected, mutually agreeable sampling locations to evaluate water quality beyond the point source outfall. The monitoring locations are coordinated with the State's existing ambient and biological monitoring networks, to provide a more comprehensive picture of watershed conditions without duplicating efforts. More information can be found here: https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/ecosystems-branch/monitoring-coalition-program

Notes:

Mention of trade names or commercial products in this document does not constitute endorsement or recommendation for use by the North Carolina Division of Water Resources.

The rules and regulations of the NC Wastewater/Groundwater Laboratory Certification Program (WW/GW LC) have primacy over this guidance document. In the event any discrepancies are noted, follow the requirements of the WW/GW LC and notify the DWR coalition coordinator so that the discrepancy may be addressed.

Acronyms/Abbreviations

Acronym or Abbreviation	Meaning
40 CFR 136	Code of Federal Regulations, Title 40, Part 136
AMS	Ambient Monitoring System
ATC	Automatic Temperature Compensation
DEQ	Department of Environment Quality
DO	Dissolved Oxygen
DWR	Division of Water Resources
EPA	Environmental Protection Agency
GPS	Global Positioning System
ISU	Intensive Survey Unit
KCl	Potassium Chloride
MOA	Memorandum of Agreement
MSDS	Material Safety Data Sheet
NaCl	Sodium Chloride
NPDES	National Pollutant Discharge Elimination System
NIST	National Institute of Standards and Technology
QA	Quality Assurance
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
SM	Standard Methods
SOP	Standard Operating Procedure
TKN	Total Kjeldahl Nitrogen
TP	Total Phosphorus
USGS	United States Geological Survey
WW/GW LC	NC Wastewater/Groundwater Laboratory Certification Program

Guidance Document Revision Log

Date	Version	Chapter	Changes	Name
11/13/2017	2.1	Entire	Updated Names and Titles	David Huffman
11/9/2017	2.1	V	Added I. Phytoplankton and J. Cyanotoxin	David Huffman
7/9/2012	2.0	Entire	Removed "NPDES" and "Discharge" from Program title	Carrie Ruhlman
7/9/2012	2.0	Entire	Updated name of DWQ Wastewater/Groundwater Laboratory Certification Program (WW/GW LC) and dates of applicable lab documents	Carrie Ruhlman
7/9/2012	2.0	I	Updated DWQ ISU SOP Manual and added website	Carrie Ruhlman
7/9/2012	2.0	II	Added recommendation to park out of the flow of traffic; removed flashing beacon statement; removed language about MSDS	Carrie Ruhlman
7/9/2012	2.0	III	Added requirement to record field data in dedicated notebook	Carrie Ruhlman
7/9/2012	2.0	III.A.1	Updated language; removed requirement to sample on upstream side of bridges and suggested using best professional judgment; changed notation requirement to locations other than those specified in MOA; specified extended period of time as greater than 3 months	Carrie Ruhlman
7/9/2012	2.0	III.A.2	Changed GPS verification requirement to the Coalition's most	Carrie Ruhlman

Guidance Document Revision Log

Date	Version	Chapter	Changes	Name
			recent MOA; added requirement to record and verify location at all new stations	
7/9/2012	2.0	III.A.4	Updated to require ten-day interval between ALL sampling events within and between months.	Carrie Ruhlman
7/9/2012	2.0	III.A.5	Added laboratory or coalition may notify coalition coordinator about missed samples; added "puddles" to list of waters not to be sampled; added instructions for 2x/month summer sampling	Carrie Ruhlman
7/9/2012	2.0	III.B.1	Removed requirement to sample on upstream side of bridge; added no wading during high flow events	Carrie Ruhlman
7/9/2012	2.0	III.B.2	Added requirement to use WW/GW LC approved methods for field parameters and QA policies (2/10/2012); removed sampling under low light conditions with supplemental lighting; added provision not to sample disconnected pools or puddles of water	Carrie Ruhlman
7/9/2012	2.0	III.B.3	Added "take care to avoid dumping preservative out"	Carrie Ruhlman
10/2/2012	2.0	IV.B.2	Removed annual temperature verification for DO meter – no longer required by WW/GW LC	Carrie Ruhlman
7/9/2012	2.0	IV.D	Added a pH field meter and probe to monitor coalition sites	Carrie Ruhlman

Guidance Document Revision Log

Date	Version	Chapter	Changes	Name
10/2/2012	2.0	IV.D.1	Removed annual ATC verification for pH – no longer required by WW/GW LC	Carrie Ruhlman
7/9/2012	2.0	IV.E.3	Removed language about regular lab certification calibration under "Note"	Carrie Ruhlman
7/9/2012	2.0	IV (throughout)	Removed requirement to explain extent of calibration drift in comment section of data submittal sheet	Carrie Ruhlman
7/9/2012	2.0	V.A.1	Removed reference to website for preservation guidelines and added reference to Appendix H; Added using fresh ice throughout day	Carrie Ruhlman
7/9/2012	2.0	V.B	Added recommendation discouraging photic zone samples from bridges; removed details of collection method and referred to website	Carrie Ruhlman
7/9/2012	2.0	V.D	Added enterococci; referenced webpage for reporting requirements; removed sample collection guidelines	Carrie Ruhlman
7/9/2012	2.0	V.E	Removed general requirements for collecting nutrient samples	Carrie Ruhlman
7/9/2012	2.0	V.F	Removed all metals sampling information and added info on suspension and revisions to section	Carrie Ruhlman
7/9/2012	2.0	V	Removed general requirements for collecting samples	Carrie Ruhlman
7/9/2012	2.0	V	Removed requirements for collecting samples	Carrie Ruhlman

Guidance Document Revision Log

Date	Version	Chapter	Changes	Name
7/9/2012	2.0	VI.D	Added that coolers used for preservation should be clean and in working condition	Carrie Ruhlman
7/9/2012	2.0	VI.F	Added section about sample transfer containers	Carrie Ruhlman
10/2/2012	2.0	VII.B	Added requirement for certified field labs to partake in annual blind performance test for pH and conductivity	Carrie Ruhlman
7/9/2012	2.0	VII.C	Added section on erroneous data	Carrie Ruhlman
10/2/2012	2.0	VIII	Updated references and hyperlinks	Carrie Ruhlman
7/9/2012	2.0	Appendix B	Updated data remark code table	Carrie Ruhlman
7/9/2012	2.0	Appendix C	Updated AMS field calibration sheet	Carrie Ruhlman
7/9/2012	2.0	Appendix D	Added note to obtain electronic copy of chart	Carrie Ruhlman
7/9/2012	2.0	Appendix H	Added WW/GW LC Surface Water Protection Preservation and Hold Time table	Carrie Ruhlman
7/9/2012	2.0	Appendix I	Updated Field Monitoring Checklist	Carrie Ruhlman

I. SCOPE AND APPLICATION

This document provides guidance for the collection of field data and laboratory samples for the North Carolina Monitoring Coalition Program ("coalition program"). Data collected and reported under the coalition program are useful in many areas of water quality management and decision-making, including watershed assessments, modeling, and permit writing. The purpose of this document is to offer guidelines to provide consistency, improve data quality, and maximize the usability of the data collected under the coalition program.

The Memorandums of Agreement (MOA) between each coalition and the Division of Water Resources (DWR) specify that sample collection shall be performed by trained personnel in accordance with the Monitoring Coalition Program Field Collection Guidance document. This guidance document is based on a DWR standard operating procedures manual (NC DWR, 2013)[1] that has been customized to meet the specific needs of the coalition program. These guidelines are intended to supplement the requirements and recommendations of the North Carolina (NC) Wastewater/Groundwater Laboratory Certification Program (WW/GW LC)[2] as well as to recommend pertinent practices employed by the DWR Ambient Monitoring System (AMS) Program[3]. In cases where the techniques of the AMS exceed the requirements of the WW/GW LC, the AMS techniques are highly recommended to ensure comparability between coalition and DWR data.

Each coalition has the responsibility of managing the collection, analysis, and submittal of data as described in its MOA. All field analyses and laboratory analyses must be performed by a WW/GW LC certified laboratory. Any contract laboratory hired by the coalitions to meet the MOA requirements will have their own standard operating procedures (SOP). This guidance document is to assure that the data collected by the

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[1] Intensive Survey Unit Standard Operating Procedures Manual: Physical and Chemical Monitoring, Version 2.1, December 2013 https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/intensive-survey-branch

[2] Field Parameters/Methods for which Certification is Offered. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch

[3] Ambient Monitoring System (AMS) https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/ecosystems-branch/ambient-monitoring-system

coalitions are comparable to the data collected by DWR. Consistency with DWR data enables the coalition data to be used for water quality management and decision-making. Inconsistencies from DWR techniques may prevent the application of the coalition data in certain water quality management decisions. Sampling methods and techniques not described in this document may be negotiated if they meet the requirements of the MOA, the WW/GW LC, and 40 CFR 136. Approval by DWR of any negotiated methods will be documented in writing.

II. SAFETY

Safety should be a primary concern during all monitoring activities. Laboratories conducting water quality monitoring are encouraged to document and follow their safety practices and guidelines.

Recommended safety practices include but are not limited to:

- Adhere to all applicable NC traffic regulations.
- Be aware of surrounding conditions: traffic, weather, bank stability, and environmental issues (plants, insects, animals, etc.) that can result in unsafe conditions. Take appropriate precautions.
- Park in a safe location out of the flow of traffic. Use flashers, road cones and/or vehicle strobe to warn oncoming traffic when parked on the roadside.
- Monitors are advised to wear a reflective safety vest while sampling.
- Sampling on a boat should be conducted with at least two people present. Monitors should wear personal floatation devices at all times while on the boat.
- Chemicals should be stored in a safe, secure manner. Monitors are encouraged to wear safety glasses and gloves when handling chemicals.
- If a monitor or other project staff feels a station is unsafe, they should notify the coalition coordinator so alternate locations can be identified.
- Monitoring staff should never wade into the water during high flows.

III. WATER QUALITY SAMPLING OVERVIEW

The purpose of collecting water quality samples is to obtain a representative portion of the water body being evaluated. Proper sampling procedures are extremely important. Valid results and interpretations of the collected data depend on:

- Collecting representative samples;
- Employing proper sampling, handling, and preservation techniques;
- Properly identifying the collected samples and documenting collection in permanent field records (dedicated notebook);
- Maintaining the integrity of all collected samples by properly packing and transporting them to the laboratory for analysis.

A. SAMPLING LOCATION AND FREQUENCY

1. Sampling Location

Monitoring locations are fixed stations (specific latitude/longitude) chosen for specific reasons. The MOAs describe the latitude, longitude, site location, and sampling frequency for each location. Field monitoring staff should make all reasonable attempts to:

- Conduct sampling and monitoring as consistently as possible at the same location to reduce unknown sources of variation.
- Sample in the main stream channel in an area of well-mixed flow outside of any discharge mixing zones.
- Use best professional judgment to sample from a fixed location that will introduce the least amount of contamination to the sample.
- Follow the "Bank/Dock Sampling" guidelines (page 7) if site conditions require that sampling occur from these locations.

- Notify the coalition coordinator of any sites that are routinely sampled from locations other than those specified in the MOA (i.e., bank or boat dock).
- Document temporary changes to sampling locations caused by safety concerns, accessibility, and stream flow patterns, on the field sheet and in the comments section of the monthly data submittal sheet.
- Inform the coalition coordinator within one week if sampling is expected to be prevented for an extended period (greater than 3 months), or if a change in location is required for safety or accessibility reasons.

2. Site Verification

At least once per year, record GPS coordinates at each site in decimal degrees to at least the fourth decimal place (DD.DDDD). Verify the GPS coordinates against the most recent MOA.

During the first sampling event at a new site, monitors are expected to record and verify GPS coordinates against station information in the MOA.

On their first site visits, new monitors are expected to confirm the field coordinates at each sampling location with a GPS device. GPS coordinates are expected to match the coalition's MOA to at least the first three decimal places.

3. Trespassing

No trespassing is permitted on private property when accessing coalition sampling sites. Permission from the property owner must be obtained for each coalition station located on private property prior to sampling. Any new sites established on private property will require the monitoring coalition coordinator to gain permission from the property owner. If at any time permission to access private land is denied, prohibited, or becomes overly burdensome, the coalition must discontinue sampling and notify the coalition coordinator immediately. If access to a site requires parking in a private driveway, the monitor must obtain permission from the property owner.

4. Sampling Frequency

Sampling must be performed in accordance with the schedule described in each coalition's MOA. The DWR expects a ten-day interval between all monitoring events. This provides a better representation of water quality changes. For instance, if the June monitoring event occurs on June 28th, the July sample should be collected no sooner than July 8th unless approved by the coalition coordinator. Contract labs should

schedule events in advance to avoid missed samples or violation of this schedule during months with two field events (May – September). Should extenuating circumstances exist, in which the lab cannot meet the ten-day interval, the coalition coordinator should be contacted to discuss alternative options.

5. Missed Samples

Every reasonable attempt should be made to sample in accordance with the schedule in the MOA. Sampling may be temporarily prevented at times due to site inaccessibility (flooding, bad weather, road construction, etc.), drought, instrument failure, or other unforeseen events. In these cases, the monitor, laboratory, or coalition is expected to notify the coalition coordinator within one week. Additionally, justification for the missed sample must be provided in the comments section of the monthly data submittal sheet.

- In the case of instrument failure, it is expected that the monitor will have backup equipment or will return to the site and sample as soon as possible. Instrument failure is not an acceptable reason for missing a monthly sampling event.
- If the sampling event is prevented due to a long-term disturbance, such as bridge construction, the coalition coordinator should be notified and will determine if site relocation is necessary.
- Sampling may be prevented due to dry stream conditions. Sampling should be suspended when the stream consists of stagnant, disconnected pools or puddles of water with no observable hydrological connection. Samples should not be collected when there is not enough volume in the stream to sample without collecting bottom sediment and/or surface scum. The monthly data submittal sheet must include the date, station number, and comment "dry stream" if these situations are encountered. If the sample is collected under extremely low flow conditions, it is recommended that a comment be made in the comments section of the monthly data submittal sheets.

If a station is required to be sampled twice monthly during May through September, the monitor should be prepared to collect required samples during either the first or second sampling event of the month. If samples cannot be collected during either event, justification must be provided.

B. FIELD SAMPLING TECHNIQUES

1. Bank/Dock Sampling

Although sampling at bridge locations is preferred, sample collection must sometimes occur from a streambank or boat dock. Bank and dock sampling require special considerations to obtain representative samples. An extension pole, wading, or a combination of wading with an extension pole may be required. Perform dock sampling on the end of the dock on the upstream side.

Extension Pole: An extension pole allows the field probe and/or sampling bottle to reach as far into the center of the main flow as possible. When collecting samples, orient the pole sampler so that the mouth of the collection bottle faces upstream. Submerge the mouth of the bottle downward to avoid collecting surface scum.

Wading: Enter downstream of the sampling location and wade upstream. Disturb the sediment as little as possible, especially in slow moving waters or in areas of fine sediment. Samples must be collected upstream of the monitor. An extension pole may be needed when wading to extend the sampling bottle to reach midstream conditions. For safety reasons, monitors should not wade into waterbodies during high flow events.

2. Field Parameter Measurement Technique

Field parameters are generally measured directly in the water with a multi-parameter meter. This section describes general guidelines when using field meters to take field parameter measurements. Information regarding field meter calibration, calibration verification, data validation and reporting and maintenance is described in the section on "Field Parameters" beginning on page 18.

Methods approved by the WW/GW LC[4] must be used to measure field parameters.

Field parameters measured by the coalition program include:

- conductivity (specific conductance at 25 °C in µS/cm)
- dissolved oxygen (DO, reported in mg/L)

[4] Field Parameters/Methods for which Certification is Offered. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch

- pH (SU)
- temperature (°C)

Temperature must be measured immediately. Dissolved oxygen and pH must be measured within 15 minutes of sample collection, but it is recommended that these be measured directly in the stream. Conductivity has a holding time of 28 days; therefore, it may be measured in the field or in the lab (see the section "Conductivity (Specific CONDUCTANCE at 25 °C)" on page 29). It is preferred to measure these four field parameters directly in the stream.

Observe the following guidelines for field parameter measurements:

- Adhere to all NC WW/GW LC Approved Procedures for Field Analysis[5].
- Calibrate the meter prior to each sampling event following the guidelines in the appropriate subsections in "Field Parameters" beginning on page 18.
- Submerge probes completely below the surface. Do not allow the probe to disturb the sediment or lay on the stream bottom.
- Ambient light must be great enough to visibly observe the location of the probe and stream conditions.
- Flow should be sufficient for recording accurate dissolved oxygen readings.
- Disconnected pools or puddles of water should not be sampled.
- Readings should be given sufficient time to stabilize (typically at least one minute) before values are recorded.
- Any unusual readings should be confirmed by checking the probe in a standard that will bracket the field reading.
    - NOTE: pH 2 or 12 buffer may be needed for extreme situations. For instance, when monitoring in certain swamp waters that frequently have pH readings less than 4, it is recommended to confirm the pH probe response in pH 2 buffer.

[5] NC WW/GW LC Approved Procedures for Field Analyses. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies

- Final calibration verifications for each parameter (see the section "Calibration Verification and Data Validation" on page 19) must be performed and recorded at the end of the sampling event to verify the meter did not drift out of acceptable calibration limits.

3. Grab Samples

Grab samples are used to characterize the water at a particular point in time. All grab samples must be taken at approximately 6 inches (0.15 m) below the surface unless otherwise requested. Grab samples at a site should also be collected over a period of time not exceeding 15-minutes, in accordance with the DWR Intensive Survey Unit SOP (DWR, 2013)[6]. Collect grab samples in the same location where field parameters are taken. This should be in a region of well-mixed flow, as described in the section on "Sampling Location", page 4). It is recommended that monitors wear a new pair of disposable laboratory gloves at each site to avoid sample contamination.

The following parameters are to be collected as grab samples in the coalition program, unless otherwise specified:

- Fecal coliform
- Nutrients: ammonia (NH_3); total Kjeldahl nitrogen (TKN); nitrate/nitrite (NO_3+NO_2); total phosphorus (TP)

  NOTE: *At sites where chlorophyll a is collected as a photic zone sample, nutrients will also be collected as a photic zone sample. At sites where chlorophyll a is sampled in summer months only, nutrients are to be collected as a photic zone sample year-round.*

- Suspended residue (a.k.a. total suspended solids; TSS)
- Turbidity
- Metals: aluminum (Al), arsenic (As), cadmium (Cd), copper (Cu), chromium (Cr), iron (Fe), mercury (Hg), manganese (Mn), nickel (Ni), lead (Pb), zinc (Zn)

[6] Intensive Survey Unit Standard Operating Procedures Manual: Physical and chemical Monitoring, Version 2.1, December 2013. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/intensive-survey-branch

GRAB SAMPLING PROCEDURE — DIRECT SAMPLING METHOD

1.) Always ensure bottle labels match the sampling site location.

2.) Remove the cap from the bottle just before sampling.

Protect the bottle and cap from contamination. Avoid touching the inside of the bottle or cap. If the inside of the bottle or cap is touched, use another one. If the cap must be set down, place it so that the inside of the lid faces upward.

3.) Plunge the bottle into the water with the mouth facing downward and pointing upstream while avoiding surface scum (Figure 1).

Take care to avoid dumping preservative out. The mouth should also be oriented away from the hand of the collector, the shore, the side of the sampling platform, or the boat.

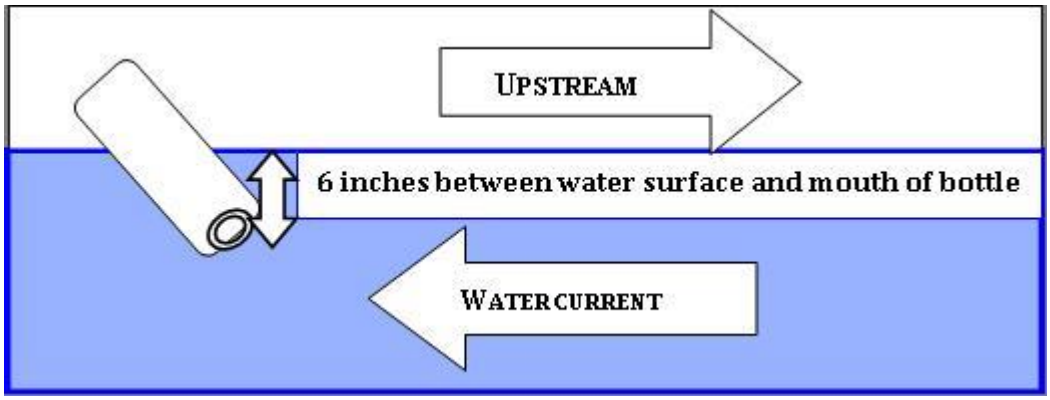

Figure 1 Grab sample bottle orientation

4.) Once underwater, tip the bottle slightly upwards to allow air to exit and the bottle to fill.

The mouth of the bottle should be approximately 6 inches (0.15 m) below the water surface.

5.) Once full, recap the bottle.

Unless otherwise specified by the analytical laboratory, leave a small headspace. This allows the sample to be shaken prior to analysis.

6.) Add preservatives if needed.

7.) Ensure the bottle is labeled appropriately.

Check that the following information is correct: site location, date, time, collector, parameter(s), and preservative(s).

8.) Place bottles in a cooler, on ice, within 15 minutes for transport to the lab.

*Follow the guidelines in the section "*Bottles and Preservation*" (page 32) regarding thermal preservation of samples on ice.*

GRAB SAMPLING — INTERMEDIATE SAMPLING DEVICES

Sampling from bridges may require the use of an intermediate sampling device.

Intermediate sampling devices include cage samplers (Figure 2), pole samplers (Figure 3), weighted bottle frames, and similar custom-made devices. Custom-made sampling devices should be evaluated by the DWR coalition coordinator prior to use. The container employed as the intermediate sampling device should be constructed so that it meets the needs of the parameter(s) to be investigated.

Figure 2. Cage sampler

Figure 3. Pole sampler

GRAB SAMPLING INTERMEDIATE DEVICES TECHNIQUE

1.) Always ensure bottle labels match the sampling site.

2.) Place the bottle(s) securely in the intermediate sampling device.

Confirm that each bottle is held securely.

3.) Remove the bottle cap(s) and lower the device to the water.

Protect the inside of the bottle and cap from contamination. Avoid touching the inside of the bottle or cap. If the inside of the bottle or cap is touched, use another one. If the caps must be set down, place so that the inside of the lid faces upwards.

4.) Swing the sampling device downstream and then allow it to drop into the water while pulling on the rope so as to position the bottle openings upstream.

Take care not to disturb the bottom sediment.

5.) Allow bottle(s) to fill.

6.) Pull the sampling device out of the water.

7.) Take care not to dislodge dirt or other material from the sampling platform.

8.) Unless otherwise specified by the analytical laboratory, leave a small headspace; this allows the sample to be shaken prior to analysis.

9.) Add preservatives if needed.

10.) Recap the bottle(s), remembering not to touch the inside of the bottle or cap.

11.) Ensure each bottle is labeled appropriately.

Check that the following information is correct: site location, date, time, collector, parameter(s), and preservative(s).

12.) Place bottles in a cooler, on ice, within 15 minutes for transport to the lab.

*Follow the guidelines in the section "*Bottles and Preservation*" (page 32) regarding thermal preservation of samples on ice.*

4. Photic Zone Sampling

Photic zone samples, also known as depth-integrated composite samples, are collected in the portion of the water column where photosynthesis by phytoplankton occurs. The photic zone is defined by the DWR as twice the Secchi depth (described in Secchi Depth Measurements, page 14). Photic zone samples are collected by lowering an integrated depth-sampling device, such as a Labline® sampler (Figure 4) to twice the Secchi depth and then slowly raising the device to the surface to obtain a representative water sample. The sampler is raised and lowered at a slow, constant pace throughout the region of twice Secchi depth until the sampler is full.

Photic zone samples are collected for:

- Chlorophyll *a* samples (at designated sites, assuming adequate water depth)
- Nutrients (to be collected as photic zone samples at sites where chlorophyll *a* is collected as photic zone samples. Nutrients should be collected year-round as photic zone samples even when chlorophyll *a* is only collected in the summer months)

Figure 4. Labline® sampler for photic zone measurements

Photic Zone Sampling Technique

To collect a vertical spatial composite sample in the photic zone:

1.) Measure and record the Secchi depth (See "Secchi Depth Measurements", page 14).

2.) Prior to collecting sample for analysis, rinse the integrated sampling device (Labline®) with sample water:

   a. Lower the Labline to the water. Take care not to disturb sediment.

b. Fill the Labline with water from the photic zone.

c. Raise to surface, swirl, and pour out water. Pour water away from sampling location so that the rinse water will not disturb the sample. After rinsing, collect the sample for analysis.

3.) Lower the Labline to twice the Secchi depth. (i.e. if the Secchi depth is 0.5 m, lower the Labline so that the fill hole on the sampler is at 1 m depth).

4.) Raise the sampler to the surface at a slow, constant rate to collect a representative sample of the water column in the photic zone.

5.) Continue to lower and raise the Labline throughout the photic zone in a slow, constant motion until the sampler is full. Maintain a steady pace to ensure a representative sample.

6.) Pour sample from Labline into appropriate sample bottle.

NOTE: *Chlorophyll a sample bottles must protect the sample from light. Brown opaque bottles are commonly used.*

7.) Rinse Labline thoroughly with distilled water after using to clean between sites.

Directions for cleaning the Labline® are found in Labline® samplers*, page 38).*

5. Secchi Depth Measurements

A Secchi disk with alternating black and white quadrants is used to measure water clarity (Figure 5). The disk is lowered into the water until it is no longer visible. The disk is then raised until it reappears. The Secchi depth is the average of the depths at which the disk vanished and reappeared.

<u>Secchi depth measurements are used to:</u>

- determine the photic zone depth
- quantify water clarity and to give a general indication of problems with algae, zooplankton, water color, and silt

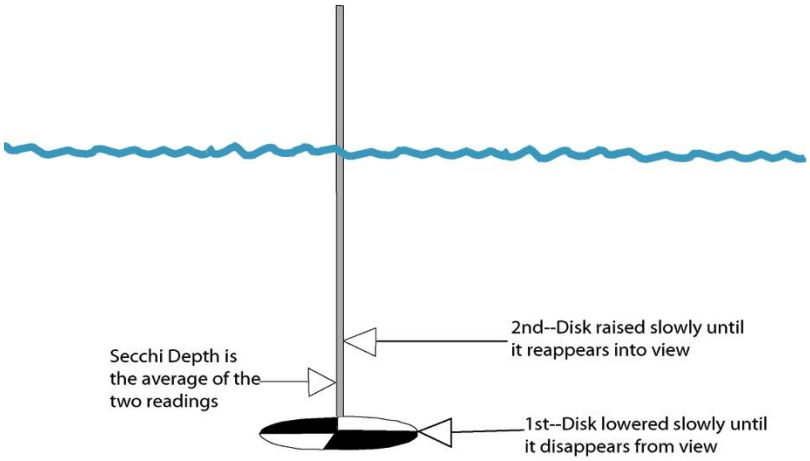

Figure 5. Secchi depth measurement

Secchi Depth Measurement Technique:

1.) Attach the Secchi disk to a rope or steel rod accurately graduated in tenth meter increments.

    NOTE: *Verification of rope graduation against a meter stick is necessary as rope will shrink and/or stretch with continued use. Additionally, markings may fade over time and be hard to distinguish. Verify at least once quarterly.*

2.) Position yourself with the sun at your back, on the shaded side of a vessel, bridge or dock.

    Do not wear sunglasses.

3.) Slowly lower the Secchi disk into water until it just disappears.

4.) Read the depth at which it disappeared from the marked line.

5.) Raise the Secchi disk until it re-appears.

6.) Read the re-appearance depth from the marked line

7.) Take the average of the two readings to get the limit of visibility.

8.) Record the average reading on field sheet as Secchi depth reading to the nearest tenth of a meter.

TIP: *In waters with strong current, the use of a graduated steel rod rather than a rope is recommended. An extra weight may need to be applied to the disk so that it remains flat and level under water*.

Reporting Secchi Depth Measurements

Report the Secchi depth measurement in the comments section of the monthly data submittal sheet to the nearest tenth of a meter.

C. QUALITY CONTROL (QC) SAMPLES

Certain procedures or special studies may require the collection of QC samples. The need and types of QC samples will be discussed with and documented by the coalition coordinator. The definitions below are provided for clarity when discussing various QC samples.

Split Sample: A split sample is a sample that has been equally divided into two or more subsamples. If multiple samples must be collected from the stream in order to gather an adequate sample volume for the split, mix all samples into one container and add any preservatives required. Then, split the homogenized, preserved sample into subsamples. Split samples are normally used to estimate variability introduced during sample transportation, processing, and laboratory analysis.

Duplicate Sample: Duplicate samples are two or more samples collected simultaneously from the same source and under identical conditions into separate containers (i.e. two grab samples taken side-by-side into two discrete containers). Duplicate samples are normally used to measure the natural variability of the sampling medium as well as the precision of a method, monitor, and/or analyst.

Field Blank: Field blanks are used to determine whether contamination has been introduced during sampling, storage, transport, and lab analysis. Field blanks are prepared in the field by processing analyte-free water through each of the steps involved in sampling. Field blanks should be prepared before collecting and processing the environmental samples at the site. For instance, analyte-free water is taken into the field in a carboy and poured into a sample container, which is then capped and transported to the lab like all other samples. If the

method calls for the addition of a preservative, this should be added to the field blank in the same manner as the other samples. Field blanks are submitted to the lab along with all other samples.

Trip Blank: Trip blanks are prepared in the lab. They are samples of analyte-free media collected in the same type of container as the environmental sample, transported from the laboratory to the sampling site, and returned to the laboratory unopened. A trip blank is used to determine if contamination occurred due to improper sample container cleaning, contaminated blank source water, or exposure to contaminants during sample storage or transport.

Temperature Blank: A temperature blank is a container of water that is shipped with field samples and is used to determine whether the samples have been adequately cooled during shipment and storage. Temperature blanks allow a representative temperature to be taken without the risk of contaminating the samples. One temperature blank should be used per cooler. Temperature blanks can be prepared at any time before or during field sampling activities. Temperature blanks should be handled as environmental samples, and should be clearly labeled as "temperature blanks". Under no condition should a thermometer or other temperature measuring device be placed in an environmental sample container to document temperature. Additional guidance on thermal preservation requirements can be found in the NC DWR Laboratory Section Sample Submission Guidance Document (DWR, 2016)[7].

[7] NC DWR Laboratory Section Sample Submission Guidance Document, November 2016. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/sample-submission#Submission

IV. FIELD PARAMETERS

A. FIELD PARAMETER MEASUREMENTS OVERVIEW

This section describes meter calibration, calibration verification, data validation and reporting, and meter maintenance. Field parameters include:

- conductivity (specific conductance at 25°C, reported in µS/cm to the nearest whole number)
- dissolved oxygen (DO, reported in mg/L to the nearest tenth)
- pH (reported in SU to the nearest tenth)
- temperature (reported in °C to the nearest tenth)

NOTE: *The section"* Field Parameter Measurement Technique*" beginning on page 7, describes techniques when using meters to collect field parameter data at the sampling location.*

1. DWR Certification

Contract labs are required to be certified by the DWR WW/GW LC. Field parameters may be certified under either laboratory certification or field parameter certification. Laboratory certification enables a contract lab to make field parameter measurements in the lab or in the field. DWR WW/GW LC rules are found in 15A NCAC 02H.0800[8]. The Approved Procedures for Field Analyses (DWR, 2012)[9] must be adhered to.

2. Field Instruments

A wide array of instruments from numerous manufacturers are available for collecting field parameter data. As a result, detailed instructions cannot be provided. Follow the

[8] 15A NCAC 02H.0800. https://deq.nc.gov/document/labcertificationrule15ancac2h0800highlighted-20090901-dwq-lab-cert-lab-certification-rule

[9] NC WW/GW LC Approved Procedures for Field Analyses. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies

manufacturer's directions for instrument calibration, use, maintenance, and storage. The guidelines in this document are supplemental to and not a replacement for the manufacturer's directions and DWR WW/GW LC rules and policies.

3. Field Meter Calibration

Meters must be calibrated prior to each sampling event for DO, pH, and conductivity. All pre-and post-calibration data must be recorded on a standardized calibration sheet. Pre-calibration data that differs greatly from post-calibration data may be a useful indicator that a meter problem exists or is developing and may help to pinpoint the source of the problem.

EXAMPLE: *An extremely low % DO saturation prior to calibration, such as 65%, often indicates the membrane on the DO probe needs to be changed. In some cases, a great variation in pre-calibration data from the expected calibration value may prevent calibration entirely. For instance, a YSI polarographic DO probe will generally not calibrate if it reads less than 50-60% saturation prior to calibration.*

4. Calibration Verification and Data Validation

In order to verify and validate the data, a post-sampling calibration verification must be performed at the end of each sampling run to assess the extent of calibration drift for pH, conductivity, and DO. A mid-day calibration verification is also recommended. Additional calibration verifications may be needed if unusual or impossible readings are observed (i.e. pH reading of 17). The requirements for field meter calibration and verification can be found on the WW/GW LC website[10].

Calibration Verification Technique

To perform a calibration verification, follow the same steps in the calibration routine without recalibrating the meter. When the probe(s) are under stable calibration conditions, record the time and the meter readings. Compare the meter readings to the expected standard values. The difference between these readings is the calibration drift. To achieve consistency with DWR programs and ensure the maximum usability of the data in water quality management areas, the maximum amount of allowable drift is specified in the Approved Procedures for Field Analyses documents[10]. These criteria

[10] NC WW/GW LC Approved Procedures for Field Analyses. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies

are presented in Table 1. Re-calibration is required if the meter exceeds the maximum amount of allowable drift. If performing the calibration verification in more than one standard, check all standards before recalibrating.

Table 1. Recommended calibration drift acceptance criteria

Parameter:	Maximum amount of acceptable drift used by DWR AMS:
pH	± 0.1 pH units from pH buffer value
Specific Conductance (at 25 ° C)	± 10% from standard value
Dissolved Oxygen (DO)	± 0.5 mg/L from theoretical DO value (from DO solubility tables)

Calibration verification is necessary for determining whether the instrument was within acceptable calibration throughout period in which samples were analyzed. A failed calibration verification indicates that the initial calibration is no longer valid and the meter requires recalibration (assuming the calibration verification was performed properly). All samples taken between the calibration and the failed verification must be flagged with a data remark code of J12 ("the calibration verification did not meet the calibration acceptance criterion for field parameters") on the monthly data submittal sheet. For calibration verifications involving more than one standard, the data must be flagged if the verification fails in any of the standards. Appendix A – Flagged Data (page 46) contains an example data submittal utilizing the J12 remark code and calibration drift documentation. Appendix B – Data Remark Codes (page 47) presents the data remark codes used in DWR programs. These codes can also be found on the Laboratory's Sections Quality Assurance website[11].

The DWR AMS calibration/calibration verification sheet is presented in Appendix C (page 53) for an example of how DWR incorporates calibration verifications into the calibration process.

The data verification procedure described in this document is designed to provide comparability with DWR AMS field parameter data and to prevent the use of suspect or

[11] North Carolina Division of Water Resources (NC DWR). https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/methods-pqls-qa

erroneous data in use support and impairment decisions. Alternative methods of data validation may be acceptable. In order to ensure the maximum level of data usability, alternative methods must be discussed with the DWR coalition coordinator and acceptance documented in writing.

5. Field Meter Maintenance

Regular maintenance must be performed on all meters. All maintenance should be recorded in a dedicated maintenance log. This log will reduce the occurrence of meter failure in the field, especially when multiple personnel use the same meter.

Common indicators that maintenance is needed:

- Meter will not calibrate
- Erratic, unstable readings
- Meter drifts quickly and frequently after calibration
- Meter repeatedly fails calibration verification
- Pre-calibration values differ greatly from standards
- Slow response time (the meter takes a long time to stabilize after the instrument is turned on and/or when submersing in sample)
- Probe condition is visibly compromised
- DO membrane is wrinkled, dry, or covered with biological growth
- Discolored spots on DO anode or cathode
- pH probe has film on it or reference junction is discolored
- Dirt/debris in conductivity cell
- Record of last maintenance is not available

6. Backup Field Meter

Meter failure is expected and may occur at any time. Therefore, it is expected that personnel carry functional back-up meters, maintenance accessories, and batteries with them always. Meter malfunction is not an acceptable reason for failing to collect monthly data.

B. TEMPERATURE

Immerse the temperature sensor in water long enough to reach equilibrium (as indicated by a stable temperature). Temperature stability will vary according to water circulation at the sampling site, but is typically within ± 0.5 °C. Report reading to 0.1 °C resolution.

1. Annual Temperature Calibration Check

The DWR WW/GW LC Policy requires that all thermometers and temperature measuring devices be checked every 12 months against a NIST traceable thermometer and the process documented. This requirement is described in the approved procedure for field analysis of temperature on the DWR WW/GW LC website[12]. The serial number of the NIST traceable thermometer that is used in the comparison must be documented. The thermometer/meter readings must be less than or equal to 1°C from the NIST traceable thermometer reading. The temperature correction (even if it is zero) must be posted on the meter as well as in hard copy format.

2. Cleaning and Maintenance of Temperature Sensors

A dirty temperature sensor may cause erroneous readings. Refer to the manufacturer's instructions for cleaning and maintaining the temperature sensor.

C. DISSOLVED OXYGEN

Dissolved oxygen (DO) calibration and maintenance guidelines included in this section are to be used as a supplement to the meter manufacturer guidelines and DWR certification requirements and not as a replacement. Methods for calibration include using water-saturated air, and Winkler titrations. Because water-saturated air is the most common method, it is the sole method described in these guidelines. The DWR

[12] NC WW/GW LC Approved Procedures for Field Analysis of Temperature. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies

WW/GW LC Approved Procedure for Field Analysis of Dissolved Oxygen can be found on the DWR WW/GW LC website[13].

1. DO Temperature Dependence

DO is a temperature-dependent parameter; therefore, the temperature sensor must be working correctly and be submersed in solution. Temperature and DO values must be stable before calibrating.

2. Dissolved Oxygen Calibration in Water-Saturated Air

Good practices for water-saturated air calibration are outlined below. These are intended to supplement the manufacturer's guidelines and not to be used as a replacement.

- Allow adequate time for the meter to stabilize, generally 5-15 minutes.
- Ensure the calibration chamber is vented to the atmosphere. It is critical that the pressure within the calibration cup be equal to the ambient atmospheric pressure.
- Ensure no water droplets are on the membrane or temperature sensor during a calibration or calibration verification. If water droplets are observed on the membrane, the water droplets must be removed (by blotting or shaking the probe) and the calibration or calibration verification process must be repeated.

  NOTE: *This is a very common source of error in the DO calibration and calibration drift checks. Water droplets on the membrane typically produce low readings. Droplets on the temperature sensor cause temperature instability due to evaporation.*

- Ensure the temperature is stable (generally stable to within 0.5°C).
- Obtain accurate barometric pressure or altitude for the calibration location. Know the barometric pressure or altitude for each sampling site in the event that the meter needs to be checked or recalibrated in the field.

[13] NC WW/GW LC Approved Procedures for Field Analysis of Dissolved Oxygen, December 2012. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies

- Record the barometric pressure or altitude used in the calibration.
- Record the following readings both before and after calibration:
    - Temperature
    - DO in mg/L
    - DO % saturation (recommended for quick troubleshooting)
- Use an oxygen solubility table to verify the meter calibrated to within 0.5 mg/L of the theoretical oxygen solubility for the corresponding barometric pressure (or altitude) and temperature. Do this by comparing the meter reading after calibration to the table value. Appendix D presents the oxygen solubility table used by the DWR AMS. Oxygen solubility tables can also be downloaded from the USGS website[14].

3. DO Calibration Verification and Data Validation

In order to validate the DO readings, an end-of-day dissolved oxygen calibration verification must be performed[15]. A mid-day calibration check is also recommended.

Calibration verification follows the same steps in the calibration routine without recalibrating the meter. When the DO probe is under stable calibration conditions, record the following readings:

- Date
- Time
- DO readings in mg/L
- DO % saturation (recommended for quick troubleshooting)
- Altitude or barometric pressure at the site where the calibration verification is performed

Then, compare the meter reading to theoretical oxygen solubility values (Appendix D). Use of the recommended ± 0.5 mg/L criterion will provide consistency with other DWR programs. If the meter does not read within 0.5 mg/L of the theoretical value, recalibrate

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[14] Dissolved oxygen solubility tables http://water.usgs.gov/software/dotables.html

[15] NC WW/GW LC Approved Procedures for Field Analysis of Dissolved Oxygen, December 2012. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies

the meter. Flag data between an initial calibration and a failed calibration check with a remark code of J12 on the monthly data submittal sheet, as described in the section on Calibration Verification and Data Validation and as demonstrated in Appendix A on page 46.

4. Secondary Checks for DO

Secondary checks on DO readings include performing Winkler titrations or checking readings against another meter. These checks are not required, but may be desired to evaluate or troubleshoot the calibration and calibration verification process. If a secondary check is performed, the results should be recorded on the field data sheet and submitted in the comments section of the monthly data submittals.

Winkler Titrations: The Winkler titration is an excellent way to evaluate the performance of DO meters. The method is described in Standard Methods for the Examination of Water and Wastewater (Standard Methods 20th, 21st, and 22nd eds.).

Alternate Meter: *A* secondary meter may be used to confirm meter performance. When using two meters side by side, note that the comparability of the readings will be a combination of both the accuracy of the DO probe and the accuracy of the temperature probe on each meter.

5. DO Maintenance and Troubleshooting

Follow the manufacturer's guidelines for maintenance and troubleshooting. Appendix E provides detailed troubleshooting and maintenance tips.

D. pH

The pH calibration and maintenance guidelines included in this section are to be used as a supplement to the meter manufacturer guidelines and DWR WW/GW LC approved procedures and not as a replacement. A pH field meter and probe should be used to monitor coalition sites.

1. pH Temperature Dependence

pH is a temperature-dependent parameter; therefore, the temperature sensor must be working correctly and be submersed in solution. Temperature and pH values must be stable before calibrating. Check the manufacturer's instructions regarding temperature compensation during calibration. To avoid temperature compensation errors during

calibration, it is often preferred to calibrate pH meters at temperatures close to 25°C, such as in a laboratory environment.

2. pH Calibration

Good practices for calibration are outlined below. These are intended to supplement the manufacturer's guidelines and not to be used as a replacement.

- It is generally recommended to calibrate in pH 7 buffer first. Then calibrate in additional buffers which bracket the anticipated sample values. A two-point calibration is required by the DWR Certification Branch. A three-point calibration is recommended if samples values are anticipated to have a pH both above and below a pH of 7.
- Prior to calibration, rinse probe with a small amount of calibration buffer and discard rinse.
- Many manufacturers' instructions require that the temperature-corrected buffer value (provided on the stock bottle) be entered during calibration.

  NOTE: *This becomes particularly important when using pH 10 buffers at temperatures other than 25°C.*

- To avoid temperature compensation errors during calibration, it is often preferred to calibrate pH at temperatures close to 25°C, such as in a laboratory environment.
- Ensure both pH and temperature sensor are fully submerged. Wait for both readings to stabilize before finalizing the calibration.
- Record both the pre-and post-calibration pH and temperature readings on the calibration sheet.
- Ensure meter calibrates to within 0.1 SU of the buffer value.
- After the calibration is finalized, perform immediate calibration verification. This is typically done with pH 7 buffer and is required by the DWR WW/GW LC. The meter must read the buffer within $\pm$0.1 pH units to be acceptable.

3. pH Calibration Verification and Data Validation

To validate the pH readings, calibration verification must be performed at the end of the sampling run, per the DWR WW/GW LC approved procedure for pH[16]. A mid-day calibration check is also recommended. Verification should be performed in a pH 7 buffer and another buffer which brackets the sample values. Verifying the pH calibration in 2 buffers will provide consistency with other DWR programs and provide a higher level of confidence in the data.

To perform the check, follow the same steps in the calibration routine without recalibrating the meter. When the pH probe is under stable calibration conditions, record the following information in each buffer without recalibrating the meter.

- Date
- Time
- Buffer value
- Calibration check value

If the meter reading differs from any of the buffers by more than 0.1 SU, the meter must be recalibrated. Flag the data between the initial calibration and the failed calibration check with a remark code of J12 on the monthly data submittal sheets, as described in The Calibration Verification and Data Validation section, and as demonstrated in Appendix A.

4. pH Maintenance and Troubleshooting

Follow the manufacturer's guidelines for maintenance and troubleshooting. Appendix F provides more detailed troubleshooting and maintenance tips.

[16] NC WW/GW LC Approved Procedures for Field Analysis of pH, November 2012. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies

E. CONDUCTIVITY (SPECIFIC CONDUCTANCE AT 25 °C)

Conductivity is to be measured and reported as specific conductance at 25°C. The specific conductance calibration, calibration verification, and maintenance guidelines included in this section are to be used as a supplement to the meter manufacturer guidelines and DWR certification requirements and not as a replacement.

1. Conductivity Temperature Dependence

Accurate conductivity measurements depend on accurate temperature measurements and accurate temperature compensation. It is critical that the temperature sensor used for temperature compensation be working properly and be submerged along with the conductivity probe when calibrating and making measurements. Calibrating under controlled conditions close to 25°C will help reduce any temperature compensation errors during calibration.

Annual Automatic Temperature Compensation Verification for Conductivity

DWR WW/GW LC policy requires that the internal Automatic Temperature Compensator (ATC) be verified every 12 months and the process documented. The DWR WW/GW LC outlines the procedure to verify the ATC in the Approved Procedures for Field Analysis of Specific Conductance document (DWR, November 2012) on their website[17].

2. Conductivity Calibration Standards

Potassium chloride (KCl) conductivity standards must be used for calibration[17]. The values of the conductivity standards used to calibrate and check the meter should bracket the expected environmental sample range.

3. Conductivity Calibration

Good practices for calibration are outlined below. These are intended to supplement the manufacturer's guidelines and are not meant to be used as a replacement.

- Ensure the conductivity probe is clean and free of dirt, residue, and debris.

[17] NC WW/GW LC Approved Procedures for Field Analysis of Specific Conductance (Conductivity), November 2012. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies

NOTE: *The conductivity cell should read zero in air. If it does not, clean the cell following the manufacturer's instructions.*

- Prior to calibration or a calibration check, rinse the conductivity probe with a small amount of the calibration standard and discard rinse.
- When immersing the conductivity probe in the calibration standard, make sure the conductivity cell and temperature sensor are fully submerged and that no air bubbles are in the conductivity cell.
- Make sure "conductivity" or "specific conductance" at 25°C is selected in the calibration menu.
- Allow the conductivity and temperature readings to stabilize (typically one minute) before calibrating.
- Record both the pre-calibration and post-calibration values on the calibration sheet.
- After calibration is completed, verify the conductivity calibration in at least one other standard. Bracket the range of anticipated measurements. The conductivity reading must be within 10% of the calibration standard.

  NOTE: *Under DWR WW/GW LC Approved Procedures for Field Analysis of Specific Conductance (Conductivity)[18], one check is required after calibration and it is recommended that it be a value that will bracket the expected conductivity values of the day.*

- When performing calibration checks, it is recommended to document the acceptance range for each standard on the calibration/calibration verification sheet to make it easier for the analyst to know when the meter exceeds the acceptance range (i.e. a standard of 1000 µS/cm would have an acceptance range of 900-1100 µS/cm). An example of this can be found on the DWR AMS standard calibration/calibration verification sheet (Appendix C).

[18] NC WW/GW LC Approved Procedures for Field Analysis of Specific Conductance (Conductivity), November 2012. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies

4. Conductivity Calibration Verification and Data Validation

To validate the conductivity readings, calibration verifications must be performed at the end of a sampling run. A mid-day calibration check is also recommended. The calibration verification involves following all the same steps in the calibration routine without recalibrating the meter. When the conductivity probe is under stable calibration conditions, record the following readings:

- Date
- Time
- Conductivity Standard Value
- Probe Reading Value

To fully validate all results, the calibration check standards must bracket the range of field measurements. If the meter does not read within ±10% of the conductivity standards, the meter must be recalibrated. However, do not recalibrate until all standards are checked.

In the event of a failed calibration verification (meter reads outside of ±10% of standard value), flag the data between the initial calibration and the failed calibration verification with a remark code of J12 on the monthly data submittal sheet, as described in Section IV.A.4. Calibration Verification and Data Validation and as demonstrated in Appendix A.

5. Maintenance and Troubleshooting

Follow the manufacturer's guidelines for maintenance and troubleshooting. Appendix G provides more detailed troubleshooting and maintenance tips.

V. SAMPLE COLLECTION AND PRESERVATION

A. GENERAL

Section V contains parameter-specific guidelines for collecting water samples for laboratory analysis. All laboratory analyses must be performed at a DWR certified laboratory. The proper collection technique for each parameter may vary depending on the analytical method. Laboratory methods not prescribed by 40 CFR 136 may be used if granted alternative test procedure approval by EPA Region IV. Application of alternative test procedures should be discussed with DWR.

1. Bottles and Preservation

Appropriate bottles and preservation techniques must be used. Consult EPA 40 CFR Part 136 for the appropriate bottles and containers. Collection and preservation guidelines used by DWR staff can be found in the DWR WW/GW LC Surface Water Protection Preservation and Hold Time Table[19] in Appendix H.

Preservation on ice

Samples requiring thermal preservation must be iced within 15 minutes of sampling. Personnel should ensure that sample containers are not submerged, thereby preventing potential cross-contamination. Steps to prevent submersion of the sample containers include:

- Placing bottles upright in cooler with lids above the iceline and frequently draining melted ice water so as to ensure only solid ice is in cooler. This may require using fresh ice throughout the day.
- Placing bottles in sealed plastic bags before inserting into ice

NOTE: *Ice or ice slurry must be used for keeping samples cool. Use of frozen ice packs is not acceptable.*

[19] DWR WW/GW LC Surface Water Protection Preservation and Hold Time Table https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/sample-submission

2. Sample Documentation

Basic information required to verify sample preservation and holding time requirements include:

- Sample identification including the number and type of containers and sampling location;
- Sample collector (printed name or signature where required);
- Date and time of sample collection;
- The parameter and/or analytical method to be performed;
- Sample type (grab, composite); and
- Preservation status: temperature and chemical preservatives.

These requirements are documented in the DWR WW/GW LC Memorandum from 6/20/2007 entitled: Required Documentation for Sample Preservation and Hold Time[20].

B. CHLOROPHYLL *a*

Samples are collected as either grab samples or photic zone samples. When water depth allows, samples are typically collected as vertically integrated depth samples in the photic zone as described in Section III.B.4., Photic Zone Sampling. To collect a photic zone sample, water must be deep enough to allow a Secchi depth measurement and to allow the use of the vertically integrated sampler (i.e. Labline®) to collect water at twice the Secchi depth. Photic zone sampling from bridges is discouraged.

When water depth is not sufficient for photic zone sampling, chlorophyll *a* samples may be collected as grab samples as described in Section III.B.3., Grab Samples.

[20] DWR WW/GW LC Memorandum: Required Documentation for Sample Preservation and Hold Time, June 20, 2007. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/sample-submission

Regardless of the sampling method, all chlorophyll *a* samples must be collected into a bottle that will protect the sample from light. Brown opaque bottles (plastic or glass) are necessary. Sampling volume depends on the analytical method. Acceptable methods of chlorophyll *a* analysis can be found on the WW/GW LC website[21] under "Inorganic".

C. CONDUCTIVITY (SPECIFIC CONDUCTANCE AT 25 °C)

Conductivity is preferably measured in situ, using a field meter, but may also be collected as a grab sample for laboratory analysis. Required preservation and hold time for laboratory analysis of specific conductance can be found in the DWR WW/GW LC Surface Water Protection Preservation and Hold Time table[22] in Appendix H.

D. FECAL COLIFORM & ENTEROCOCCI

Fecal coliform and enterococci are collected as grab samples. All fecal coliform and enterococci samples must be returned to the laboratory and analyzed within eight hours from the time of collection. Required preservation and hold time information can be found in the DWR WW/GW LC Surface Water Protection Preservation and Hold Time table[22] in Appendix H. Reporting requirements can be found with the guidance documents on the DWR WW/GW LC webpage.

[21] Field Parameters/Methods for which Certification is Offered. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch

[22] DWR WW/GW LC Surface Water Protection Preservation and Hold Time Table. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/sample-submission

E. NUTRIENTS

Nutrient samples collected by the coalitions include:

- Ammonia (NH_3)
- Total Kjeldahl Nitrogen (TKN)
- Nitrate plus Nitrite ($NO_3 + NO_2$)
- Total Phosphorus (TP)

Nutrients are collected as grab samples at most sites. However, at sites where chlorophyll *a* is collected as a photic zone sample, nutrients must also be collected as a photic zone sample. At sites where chlorophyll *a* samples are taken only seasonally as photic zone samples but nutrient samples are taken year round, nutrient samples are to be taken year round as photic zone samples.

Multiple methods are approved for nutrient analysis. Follow the method guidelines regarding sample preservation. Generally, NH_3, TKN, $NO_3 + NO_2$, and TP can all be collected in one bottle and preserved with sulfuric acid to a pH less than 2. Acid may be added to the bottle before sample collection or after collection in the field. Additional information on required preservation and hold time can be found in the DWR WW/GW LC Surface Water Protection Preservation and Hold Time table[23] in Appendix H.

F. TOTAL RECOVERABLE METALS

Routine ambient data collection for total recoverable metals was suspended by the Division on April 3, 2007 via memorandum from the Division Director. For this reason, all coalitions have forgone metals monitoring. The DWR is in the process of reviewing metals water quality assessment techniques, evaluation criteria and relevant standards. Monitoring will be reinstated by coalitions at such time that the DWR concludes its review or when the Division Director mandates. An amendment detailing revisions to this section will be made to the document at that time.

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[23] DWR WW/GW LC Surface Water Protection Preservation and Hold Time Table. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/sample-submission

G. SUSPENDED RESIDUE

Suspended residue is often referred to as total suspended solids (TSS). Suspended residue is collected as a grab sample in the coalition program. Required preservation and hold time information can be found in the DWR WW/GW LC Surface Water Protection Preservation and Hold Time table[24] in Appendix H.

H. LAB TURBIDITY

Lab turbidity (also referred to as "turbidity") is collected as a grab sample in the coalition program. Required preservation and hold time information for turbidity can be found in the DWR WW/GW LC Surface Water Protection Preservation and Hold Time table[24] in Appendix H.

I. PHYTOPLANKTON

Samples may be collected as grabs, composite or scoops with sampling method written on the sample label. Only grab and composite samples may be for quantification. All samples must be preserved with Lugol's. Samples must be stored in coolers on ice at 4 C until receipt at the laboratory

J. CYANOTOXIN

Samples may be collected as grabs, composite or scoops with sampling method written on the sample label. Algae obtained for toxin analysis must be collected in PTEG or glass amber containers. Cool to 4 C. Send to the EU lab for identification prior to the Lab for analysis. Frozen samples must be analyzed within 2 weeks of collection.

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[24] DWR WW/GW LC Surface Water Protection Preservation and Hold Time Table. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/sample-submission

VI. STANDARD CLEANING PROCEDURES

All sampling equipment must be properly cleaned before sampling. Check with each analytical method for any parameter-specific equipment cleaning instructions. General recommendations include:

A. GENERAL SAMPLING EQUIPMENT

Sampling equipment should be washed with phosphate-free laboratory detergent and rinsed with hot tap water before long-term storage.

B. LABLINE® SAMPLERS

For Lablines®, clean with phosphate-free detergent and rinse at least three times with deionized water. A common practice is to cover the top of the Labline® with foil to prevent contamination and to show that the Labline® has been cleaned. Between sites, rinse with distilled water. At each site, rinse the Labline® with surface water from that site, prior to taking the sample.

C. FIELD METERS

Follow manufacturers' instructions for cleaning. Keep meter body free of dirt by wiping with a damp cloth. Rinse probes between uses and before storage. Keep probes free of dirt and debris. Store probes per manufacturers' instructions in the manufacturer provided storage cup.

D. COOLERS AND SHIPPING CONTAINERS

All ice chests and reusable shipping containers should be washed as needed with a mild, phosphate free detergent (interior and exterior), rinsed with tap water, and air dried before storage. Coolers used to preserve samples during transport should be in clean, working condition (i.e., working drain plugs and securely closing lids).

E. SAMPLING CONTAINERS

Containers that are purchased as pre-cleaned should be certified by the manufacturer or checked to ensure that the parameters tested for are below the published reporting limits. Manufacturer certificates for glass bottles should be kept on file. Containers should be stored in a manner that does not leave them susceptible to contamination by dust or other particulates and should remain capped until use. Any containers showing evidence of contamination should be discarded.

It is a good laboratory practice to periodically check bottles for contamination attributed to storage conditions at the lab by filling representative containers with analyte-free water, adding the appropriate preservatives, and submitting them to the lab for analysis. Any container lots showing analyte levels at or above reporting limits should be discarded.

F. SAMPLE TRANSFER CONTAINERS

If intermediate sampling containers are used to collect samples, those containers must be properly cleaned before use (if containers are re-used). A clean container must be used at each site.

VII. DWR FIELD VISITS AND QA/QC STUDIES

A. FIELD VISITS

At least one time per year, DWR staff will observe field sampling for each coalition, and summarize the field visit in a letter that will be sent to the coalition chairman. Field visits will involve following a monitor through a day of sampling. Visits will include review of:

- station location
- meter maintenance
- calibration
- quality assurance/quality control practices
- sampling technique
- sample preservation, handling, and transport
- safety practices
- documentation procedures

Appendix I includes an example of a field visit checklist.

B. QA/QC STUDIES

The coalition program may occasionally conduct special QA/QC studies to assess consistency of results among coalition contract labs and the DWR. These special studies may include:

- Field parameter measurement comparison using two or more field meters;
- Split sampling study in which samples for a certain parameter are split and analyzed by the DWR lab and the coalition contract lab(s). Multiple labs may be involved in the split sampling studies.

In addition, certified field labs are required to take part in an annual blind performance test for parameters for which they hold certification (excluding dissolved oxygen and temperature).

C. ERRONEOUS DATA

Should the Division or the coalitions collect or receive data of a questionable nature, it is the decision of the coalition and the coalition coordinator as to how the data will be handled. Data can be either qualified or deleted depending on the severity of discrepancies or the overall reliability of the data in question. Either way, a written request from the coalition to the Division is required to document changes to submitted data and/or justify unsubmitted results.

VIII. REFERENCES AND HYPERLINKS

A. REFERENCES

American Public Health Association. Standard Methods for the Examination of Water and Wastewater, 20th edition. Washington, D.C.

American Public Health Association. Standard Methods for the Examination of Water and Wastewater, 21st edition. Washington, D.C.

American Public Health Association. Standard Methods for the Examination of Water and Wastewater, 22nd edition. Washington, D.C.

Lewis, M. E. 2006. U.S. Geological Survey National Field Manual: Dissolved Oxygen, Version 2.1. Techniques of Water-Resources Investigations, book 9, chap A6, section 6.2: http://water.usgs.gov/owq/FieldManual/Chapter6/6.2_contents.html (last accessed 2/01/08).

North Carolina Division of Water Resources (NC DWR). Ambient Monitoring System (AMS) Quality Assurance Plan (QAPP). 2017. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/ecosystems-branch/ams-quality-assurance-project-plan

North Carolina Division of Water Resources (NC DWR). Intensive Survey Unit Standard Operating Procedures Manual: Physical and Chemical Monitoring. 2013. Version 2.1. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/intensive-survey-branch

B. HYPERLINKS

North Carolina Division of Water Resources (NC DWR) Wastewater/Groundwater Laboratory Certification Branch

https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch

North Carolina Division of Water Resources (NC DWR) Coalition Program Data Qualification Codes

https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/ecosystems-branch/monitoring-coalition-program

North Carolina Division of Water Resources (NC DWR) Laboratory Section Quality Assurance website.

https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/methods-pqls-qa

North Carolina Division of Water Resources (NC DWR) Quality Assurance Policies for Field Laboratories.

https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies

North Carolina Division of Water Resources (NC DWR) Surface Water Protection Preservation and Hold Time.

https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/sample-submission

Inorganic and Microbiological Parameters

https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/inorganic-chemistry-unit4

https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/microbiology-metals-unit

Required Documentation for Sample Preservation and Hold Time

https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/sample-submission

Standard Methods for the Examination of Water and Wastewater

http://www.standardmethods.org/

North Carolina Environmental Management Commission. 2004. State of North Carolina Department of Environment & Natural Resources, Division of Water Resources Administrative Code. Section 15A NCAC 02H.0800-Laboratory Certification Rule.

https://deq.nc.gov/document/labcertificationrule15ancac2h0800highlighted-20090901-dwq-lab-cert-lab-certification-rule

United States Environmental Protection Agency (EPA) Methods Update Rule. May 18, 2012. Federal Registrer. Volume 77, No. 97. 40 CFR Part 136.

http://www.gpo.gov/fdsys/pkg/FR-2012-05-18/pdf/2012-10210.pdf

United States Environmental Protection Agency (EPA). September 1996. The Volunteer Monitor's Guide To Quality Assurance Project Plans. EPA 841-B-96-003

http://www.epa.gov/OWOW/monitoring/volunteer/qapp/qappappa.pdf

IX. APPENDICES

APPENDIX A – FLAGGED DATA

Flagging Data taken prior to Failed Calibration Verifications

For data taken between the initial calibration and a failed calibration verification, flag data by using a "J12" in the remark code column and document the extent of calibration drift in the comments column.

J12 indicates that "the calibration verification did not meet the calibration acceptance criteria for field parameters."

The full list DWR data remark codes[25] is attached in Appendix B and is also available in the MOAs, on the DWR Laboratory Section Quality Assurance website[26] and coalition website[27].

				Temp (°C)	Temp_rmk	DO (mg/l)	DOrmk	pH (su)	pH_rmk	Conductivity (µmhos/cm)	Conductivity_rmk	See NOTE	Comments
Station	Date (m/d/yyyy)	Time (hh:mm)	Depth (m)	10.0	10 rmk	300	300 rmk	400.0	400 rmk	94	94 rmk		
A1234567	8/19/2007	15:30	0.1	25.2		7.8		6.9		133			
A9876543	8/20/2007	11:50	0.1	27.2		7.1		7.2	J12	125			
A8765432	8/20/2007	13:05	0.1	28.0		6.5		6.3	J12	122			
A7654321	8/20/2007	13:30	0.1	25.0		6.7		6.9		119			
A6543210	8/20/2007	14:50	0.1	17.0		5.5		6.7		120			
A1357924	8/21/2007	16:10	0.1	22.1		3.1		6.2		233	J12		
A0246813	8/24/2007	9:30	0.1	19.7		8.3	J12	7.0		99			
A0246813	8/24/2007	11:30	0.1	12.0		8.9	J12	7.3		115			

NOTE: *Columns on data submittal sheet between Conductivity_rmk column and Comments column not shown*

[25] NC Inorganic Branch. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch

[26] North Carolina Division of Water Resources (NC DWR). https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/methods-pqls-qa

[27] NC Monitoring Coalition Program. https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/ecosystems-branch/monitoring-coalition-program

APPENDIX B – DATA REMARK CODES

When reporting data, the DWR's data remark codes must be used to provide additional information regarding data quality and interpretation. The current set of remark codes to be used is provided below. Updates to the data remark codes can be found on the coalition program webpage[28]. Note that data remark (data qualifiers) are subject to change.

Data Remark Codes for use with Coalition Data as of 12/2011

Data Remark Code	Code Definition
A	Value reported is the mean (average) of two or more determinations. This code is to be used if the results of two or more discrete and separate samples are averaged. These samples shall have been processed and analyzed independently (e.g. field duplicates, different dilutions of the same sample). This code is not required for BOD or coliform reporting since averaging multiple dilutions for these parameters is fundamental to those methods.
B	Results based upon colony counts outside the acceptable range and should be used with caution. This code applies to microbiological tests and specifically to membrane filter (MF) colony counts. It is to be used if less than 100% sample was analyzed and the colony count is generated from a plate in which the number of colonies exceeds the ideal ranges indicated by the method. These ideal ranges are defined in the method as: FECAL COLIFORM OR ENTEROCOCCUS BACTERIA: 20-60 COLONIES TOTAL COLIFORM BACTERIA: 20-80 COLONIES B1. Countable membranes with less than 20 colonies. Reported value is estimated or is a total of the counts on all filters reported per 100 mL. B2. Counts from all filters were zero. The value reported is based on the number of colonies per 100 mL that would have been reported if there had been one colony on the filter representing the largest filtration volume (reported as a less than "<" value).

[28] North Carolina Division of Water Resources (NC DWR). https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/microbiology-inorganics-branch/methods-pqls-qa

Data Remark Code	Code Definition
B	B3. Countable membranes with more than 60 or 80 colonies. The value reported is calculated using the count from the smallest volume filtered and reported as a greater than ">" value. B4. Filters have counts of both >60 or 80 and <20. Reported value is a total of the counts from all countable filters reported per 100 mL. B5. Too many colonies were present; too numerous to count (TNTC). TNTC is generally defined as > 150 colonies. The numeric value represents the maximum number of counts typically accepted on a filter membrane (60 for fecal and 80 for total), multiplied by 100 and then divided by the smallest filtration volume analyzed. This number is reported as a greater than value. B6. Estimated Value. Blank contamination evident. B7. Many non-coliform colonies or interfering non-coliform growths are present. In this competitive situation, the reported coliform value may under-represent actual coliform density. Note: A "B" value shall be accompanied by justification for its use denoted by the numbers listed above (e.g., B1, B2, etc.). Note: A "J2" should be used for spiking failures.
BB	This code applies to most probable number (MPN) microbiological tests. 1. No wells or tubes gave a positive reaction. Value based upon the appropriate MPN Index and reported as a less than "<" value. 2. All wells or tubes gave positive reactions. Value based upon the MPN Index and reported as a greater than ">" value. Note: A "BB" value shall be accompanied by justification for its use denoted by the numbers listed above (e.g., BB1, BB2, etc.).
C	Total residual chlorine was present in sample upon receipt in the laboratory; value is estimated. Generally applies to cyanide, phenol, NH_3, TKN, coliform, and organics)

Data Remark Code	Code Definition
G	A single quality control failure occurred during biochemical oxygen demand (BOD) analysis. The sample results should be used with caution. G1. The dissolved oxygen (DO) depletion of the dilution water blank exceeded 0.2 mg/L. G2. The bacterial seed controls did not meet the requirement of a DO depletion of at least 2.0 mg/L and/or a DO residual of at least 1.0 mg/L. G3. No sample dilution met the requirement of a DO depletion of at least 2.0 mg/L and/or a DO residual of at least 1.0 mg/L. G4. Evidence of toxicity was present. This is generally characterized by a significant increase in the BOD value as the sample concentration decreases. The reported value is calculated from the highest dilution representing the maximum loading potential and should be considered an estimated value. G5. The glucose/glutamic acid standard exceeded the range of 198± 30.5 mg/L. G6. The calculated seed correction exceeded the range of 0.6 to 1.0 mg/L. G7. Less than 1 mg/L DO remained for all dilutions set. The reported value is an estimated greater than value and is calculated for the dilution using the least amount of sample. G8. Oxygen usage is less than 2 mg/L for all dilutions set. The reported value is an estimated less than value and is calculated for the dilution using the most amount of sample. G9. The DO depletion of the dilution water blank produced a negative value.
J	Estimated value; value may not be accurate. This code is to be used in the following instances: J1. Surrogate recovery limits have been exceeded; J2. The reported value failed to meet the established quality control criteria for either precision or accuracy; J3. The sample matrix interfered with the ability to make any accurate determination;

Data Remark Code	Code Definition
J	J4. The data is questionable because of improper laboratory or field protocols (e.g. composite sample was collected instead of grab, plastic instead of glass container) J5. Temperature limits exceeded (samples frozen or >6° C) during transport or not verifiable (e.g., no temperature blank provided);, non-reportable for NPDES compliance monitoring. J6. The laboratory analysis was from an unpreserved or improperly chemically preserved sample. The data may not be accurate. J7. This qualifier is used to identify analyte concentration exceeding the upper calibration range of the analytical instrument/method. The reported value should be considered estimated. J8. Temperature limits exceeds (samples frozen or >6°C during storage. The data may not be accurate. J9. The reported value is determined by a one-point estimation rather than against a regression equation. The estimated concentration is less than the laboratory practical quantitation limit and greater than the laboratory method detection limit. J10. Unidentified peak; estimated value. J11. The reported value is determined by a one-point estimation rather than against a regression equation. The estimated concentration is less than the laboratory practical quantitation limit and greater than the laboratory method detection limit. *This code is used when an MDL has not been established for the analyte in question.* J12. The calibration verification did not meet the calibration acceptance criterion for field parameters. Note: A "J" value shall not be used if another code applies (ex. N, V, M).
M	Sample and duplicate results are "out of control." The sample is non-homogenous (e.g. VOA soil). The reported value is the lower value of duplicate analyses of a sample.
N	Presumptive evidence of presence of material; estimated value. This code is to be used if: N1. The component has been tentatively identified based on mass spectral library search;

Data Remark Code	Code Definition
	N2. There is an indication that the analyte is present, but quality control requirements for confirmation were not met (i.e., presence of analyte was not confirmed by alternate procedures). N3. This code shall be used if the level is too low to permit accurate quantification, but the estimated concentration is less than the laboratory practical quantitation limit and greater than the laboratory method detection limit. This code is not routinely used for most analyses. N4. This code shall be used if the level is too low to permit accurate quantification, but the estimated concentration is less than the laboratory practical quantitation limit and greater than the instrument noise level. This code is used when an MDL has not been established for the analyte in question. N5. The component has been tentatively identified based on a retention time standard.
P	Elevated practical quantitation limit (PQL)* due to matrix interference and/or sample dilution.
Q	Holding time exceeded. These codes shall be used if the value is derived from a sample that was received, prepared and/or analyzed after the approved holding time restrictions for sample preparation and analysis. The value does not meet NPDES requirements. Q1. Holding time exceeded prior to receipt by lab
Q	Q2. Holding time exceeded following receipt by lab
S	Not enough sample provided to prepare and/or analyze a method-required matrix spike (MS) and/or duplicate (MSD).
U	Indicates that the analyte was analyzed for but not detected above the reported practical quantitation limit (PQL)*. The number value reported with the "U" qualifier is equal to the laboratory's PQL*.

Data Remark Code	Code Definition
V	Indicates the analyte was detected in both the sample and the associated method blank. Note: The value in the blank shall not be subtracted from the associated samples.
X	Sample not analyzed for this constituent. This code is to be used if: X1. Sample not screened for this compound. X2. Sampled, but analysis lost or not performed-field error X3. Sampled, but analysis lost or not performed-lab error
Y	Elevated PQL* due to insufficient sample size
Z	The presence or absence of the analyte cannot be verified. The sample analysis/results are not reported due to: Z1. Inability to analyze the sample. Z2. Questions concerning data reliability.

APPENDIX C – WATER QUALITY MONITORING FIELD METER CALIBRATION

Water Quality Monitoring Field Meter Calibration Sheet

Collector(s):

Study:

Sampling Location:

Meter Model:

Meter / Sonde Serial No:

	Date yy/mm/dd	Time 24hr hh:mm	Initials
Pre-Sampling Calibration			
Post-Sampling Check			

Barometer Calibration (mmHg)
**YSI Pro Plus Meters Only*

Initial Reading	Calibrated Value

Miscellaneous (Does not apply to YSI or Accumet Meters)

	Battery Level (V)		Stirrer Working?
Pre-Sampling Calibration			Y / N
Post-Sampling Check			Y / N

Battery Ranges – Surveyor: Internal- 7.2-7.5V, external- 11-13V; Quanta: 4.0-4.5V

Dissolved Oxygen (mg/L)

	Temp. °C	Initial % Saturation	Barometric Pressure (mmHg)	Altitude (ft.)	D.O. Table Value	Initial Meter Reading (mg/L)	Calibrated Meter Reading (mg/L)	Calibrated % Saturation
Pre-Sampling Calibration								
Post-Sampling Check								
					Within ± 0.5 ? Y / N			

Specific Conductance (µS/cm at 25°C)

Lot #: **Lot #:**

	Dry Air [1, 2] Zero (0)		Conductivity Standard [3] Value: _____		Calibration Check Value: _____
	Initial Meter Reading	Calibrated [4] Meter Reading	Initial Meter Reading	Calibrated [4] Meter Reading	Initial Meter Reading
Pre-Sampling Calibration					
Post-Sampling Check	Within ± 2? Y / N		Within ±10%? Y / N		Within ±10% Y / N

±10% Ranges for Sp. Cond.

Standard	Range
100	90 to 110
600	450 to 660
1,000	900 to 1,100
10,000	9,000 to 11,000
15,000	13,500 to 16,500
60,000	45,000 to 66,000

NOTE: Quanta reads in mS/cm; move decimal 3 places right for µS/cm.

[1] Dry Air CALIBRATIONS are conducted for 4a and M85 Hydrolabs only.

[2] Dry Air CHECKS (confirmation of zero in dry air) are conducted for YSI 85, YSI 6920, YSI Pro Plus & Quanta meters.

[3] Conductivity standards are used to CHECK the YSI 85 meter and to CALIBRATE all Hydrolab meters and the YSI 6920 & YSI Pro Plus.

[4] Does not apply to Dry Air CHECKS or Conductivity Standard CHECKS (leave blank).

pH (SU)

Lot #: **Lot #:**

	Buffer #1 7.0 Buffer Temp: _____		Buffer #2 4.0 / 10.0 Buffer Temp: _____		Slope Efficiency[5]	Confirmation Buffer 7.0
	Initial Meter Reading	Calibrated Meter Reading	Initial Meter Reading	Calibrated Meter Reading		Meter Reading
Pre-Sampling Calibration						
Post-Sampling Check	Within ± 0.2? Y / N		Within ± 0.2? Y / N			Within ± 0.1? Y / N

[5] Slope efficiency applies to Accumet meters only (does not apply to Hydrolab or YSI meters).

Comments:

Keep original on file for 5 years

Ver. 08/06/2012

APPENDIX D – DISSOLVED OXYGEN SOLUBILITY TABLE AND CORRECTION CHART

To verify dissolved oxygen reading:

- Set up probe under stable calibration conditions. When stable, record temperature reading.
- Look up temperature on chart. The corresponding DO value, given in mg/L, is the theoretical amount of oxygen that would be dissolved in water at sea level (barometric pressure = 760 mm Hg and altitude = 0 ft).
- To adjust for barometric pressure or altitude, multiply the theoretical DO value from the table by the altitude or barometric pressure correction value given in the DO correction chart on the following page.

Sea Level *(Uncorrected D.O. Values)*

Dissolved Oxygen (D.O.) TABLE

Altitude at Sea Level = 0 feet — Barometric Pressure (BP) at Sea Level = 760 mm Hg

Temp (°C)	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	Temp (°C)
0	14.6	14.6	14.5	14.5	14.5	14.4	14.4	14.3	14.3	14.3	0
1	14.2	14.2	14.1	14.1	14.1	14.0	14.0	13.9	13.9	13.9	1
2	13.8	13.8	13.8	13.7	13.7	13.6	13.6	13.6	13.5	13.5	2
3	13.5	13.4	13.4	13.4	13.3	13.3	13.2	13.2	13.2	13.1	3
4	13.1	13.1	13.0	13.0	13.0	12.9	12.9	12.9	12.8	12.8	4
5	12.8	12.7	12.7	12.7	12.6	12.6	12.6	12.5	12.5	12.5	5
6	12.4	12.4	12.4	12.4	12.3	12.3	12.3	12.2	12.2	12.2	6
7	12.1	12.1	12.1	12.0	12.0	12.0	12.0	11.9	11.9	11.9	7
8	11.8	11.8	11.8	11.8	11.7	11.7	11.7	11.6	11.6	11.6	8
9	11.6	11.5	11.5	11.5	11.4	11.4	11.4	11.4	11.3	11.3	9
10	11.3	11.3	11.2	11.2	11.2	11.2	11.1	11.1	11.1	11.1	10
11	11.0	11.0	11.0	11.0	10.9	10.9	10.9	10.9	10.8	10.8	11
12	10.8	10.8	10.7	10.7	10.7	10.7	10.6	10.6	10.6	10.6	12
13	10.5	10.5	10.5	10.5	10.4	10.4	10.4	10.4	10.4	10.3	13
14	10.3	10.3	10.3	10.2	10.2	10.2	10.2	10.1	10.1	10.1	14
15	10.1	10.1	10.0	10.0	10.0	10.0	10.0	9.9	9.9	9.9	15
16	9.9	9.8	9.8	9.8	9.8	9.8	9.7	9.7	9.7	9.7	16
17	9.7	9.6	9.6	9.6	9.6	9.6	9.5	9.5	9.5	9.5	17
18	9.5	9.4	9.4	9.4	9.4	9.4	9.4	9.3	9.3	9.3	18
19	9.3	9.3	9.2	9.2	9.2	9.2	9.2	9.1	9.1	9.1	19
20	9.1	9.1	9.1	9.0	9.0	9.0	9.0	8.97	8.9	8.9	20
21	8.9	8.9	8.9	8.9	8.8	8.8	8.8	8.8	8.8	8.8	21
22	8.7	8.7	8.7	8.7	8.7	8.7	8.6	8.6	8.6	8.6	22
23	8.6	8.6	8.5	8.5	8.5	8.5	8.5	8.5	8.4	8.4	23
24	8.4	8.4	8.4	8.4	8.4	8.3	8.3	8.3	8.3	8.3	24
25	8.3	8.2	8.2	8.2	8.2	8.2	8.2	8.2	8.1	8.1	25
26	8.1	8.1	8.1	8.1	8.1	8.0	8.0	8.0	8.0	8.0	26
27	8.0	8.0	7.9	7.9	7.9	7.9	7.9	7.9	7.9	7.8	27
28	7.8	7.8	7.8	7.8	7.8	7.8	7.7	7.7	7.7	7.7	28
29	7.7	7.7	7.7	7.7	7.6	7.6	7.6	7.6	7.6	7.6	29
30	7.6	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.4	30
31	7.4	7.4	7.4	7.4	7.4	7.4	7.4	7.3	7.3	7.3	31
32	7.3	7.3	7.3	7.3	7.3	7.2	7.2	7.2	7.2	7.2	32
33	7.2	7.2	7.2	7.1	7.1	7.1	7.1	7.1	7.1	7.1	33
34	7.1	7.1	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	34
35	6.9	6.9	6.9	6.9	6.9	6.9	6.9	6.9	6.9	6.8	35

* All D.O. values are in mg/L

Correction Factor at Sea Level = 1.0

NOTE: An electronic copy of this chart can be obtained by contacting the coalition coordinator.

APPENDIX D – DISSOLVED OXYGEN SOLUBILITY TABLE AND CORRECTION CHART, CONTINUED

Correction Chart -How to Correct D.O. Table Values

Altitude (ft)	Barometric Pressure (mmHg)	Correction Factor	Altitude (ft)	Barometric Pressure (mmHg)	Correction Factor
-300	768	1.012	2200	699	0.92
-200	765	1.008	2300	697	0.917
-100	762	1.004	2400	695	0.914
0	760	1.000	2500	692	0.910
100	757	0.996	2600	689	0.907
200	755	0.993	2700	686	0.903
300	752	0.989	2800	684	0.900
400	749	0.985	2900	682	0.897
500	746	0.981	3000	679	0.893
600	743	0.978	3100	676	0.890
700	740	0.974	3200	673	0.886
800	737	0.970	3300	671	0.883
900	735	0.967	3400	669	0.880
1000	732	0.963	3500	666	0.876
1100	729	0.959	3600	663	0.873
1200	727	0.956	3700	661	0.870
1300	724	0.952	3800	658	0.866
1400	721	0.949	3900	656	0.863
1500	718	0.945	4000	654	0.860
1600	715	0.941	4100	651	0.857
1700	713	0.938	4200	648	0.853
1800	710	0.934	4300	646	0.850
1900	708	0.931	4400	644	0.847
2000	705	0.927	4500	641	0.844
2100	702	0.924			

Corrected D.O. value = value from Sea Level D.O. Table * Correction Value

1. Use the temperature displayed on your meter and the Sea Level Table to find the Uncorrected D.O. Value
2. Use your location's altitude and the Correction Chart on this page to find the corresponding correction value.

3. Multiply the uncorrected D.O. value from step 1 by the correction value from step 2 to get the Corrected D.O. value.
4. The value calculated in step 3 (Corrected D.O. value) and the value displayed on the meter should be within +/- 0.5 mg/L of each other.

APPENDIX E – DISSOLVED OXYGEN TIPS AND DETAILS

DO Membrane and Electrolyte

Erroneous reading will result from loose, wrinkled, torn, or fouled membranes, from bubbles in the electrolyte solution, or a lack of electrolyte under the membrane. Tiny holes in the membrane may be hard to see but will negatively affect performance. The following considerations are important for DO probes:

- Visually inspect membranes prior to each use.
- No tears, bubbles, wrinkles, or biological growth (white, green, black spots or fuzz) should be observed.
- Rinse with distilled water after using to prevent biological growth.
- Store in a moist, clean environment to prevent the DO membrane from drying out.
- Between sampling sites, the probe should be placed back in the calibration cup with a small amount of moisture (moist sponge, water droplets, etc.) or wrap a damp towel around the probe guard.

Changing membranes

Changing membranes will help reduce erroneous readings. Most manufacturers recommend changing the DO membrane at least once per month. The need to change membranes depends on usage, so inspect the probe regularly. Change membranes as directed by the manufacturer. This is generally recommended in the following situations:

- When calibration is difficult or impossible
- When readings are erratic or will not stabilize
- If meter frequently fails post-sampling check
- If membrane has dried out or is damaged (i.e. tears, wrinkles, bubbles, biological growth, dirty)

Calibrating after a membrane change

Most manufacturers recommend allowing stretch-on membranes to relax overnight (6-8 hours) prior to calibration. If conditions necessitate using the sensor and new membrane before the recommended overnight conditioning time, more frequent calibration drift checks and possibly recalibration are necessary for accurate DO measurements.

NOTE: *The overnight relaxation time applies only to stretch-on membranes, not cap membranes.*

Anode and cathode condition

Clean the anode and cathode as recommended by the manufacturer. Depending on probe usage and conditions, deposits (generally black spots of silver chloride (AgCl)) will form on the anode and cathode. These deposits will reduce the sensitivity of the probe. Refer to the instruction manual or consult the manufacturer for the proper cleaning procedure. Cleaning instructions generally involve lightly buffing the gold cathode and either sanding of the silver anode with 400 grit sandpaper or soaking it in dilute ammonium hydroxide solution.

Proper Calibration and Measurement Conditions

Erroneous readings may result in the following situations:

- The probe is calibrated or the calibration drift check is performed under improper conditions, which include:
    - water droplets on the membrane or temperature sensor
    - unstable temperature (instability greater than 0.5°C)
    - pressure in the calibration cup is not equal to ambient pressure (calibration cup not vented to atmosphere)
    - Entering an inaccurate barometric pressure during calibration
    - Not having the proper altitude or barometric pressure for performing the calibration check
- The meter is used improperly when taking a measurement.

NOTE: *Sufficient water movement across the probe face is required. This is generally 6 to 12 inches unless the probe has a circulator or is engineered for reduced flow dependence*.

Dissolved Oxygen Meter Troubleshooting

Table 2 lists potential remedies for the following symptoms:

- Unable to calibrate (error message during calibration)
- Unusual or erroneous readings
- Unstable readings
- Failed calibration drift check

Table 2. Dissolved Oxygen Troubleshooting

Potential DO problem	Solution
Damaged membrane	Replace membrane with proper membrane from manufacturer
Lack of electrolyte/old electrolyte	Use fresh electrolyte. Make sure electrolyte is not expired and is the appropriate solution for the probe.
Fouled or damaged anode or cathode	Clean anode/cathode per manufacturer's instructions
Improper calibration conditions	Calibrate or verify performance under proper calibration conditions, including: • 100% humidity • stable temperature • no water droplets on membrane or thermistor • correct barometric pressure/altitude • calibration cup vented to atmosphere
Improper measurement conditions	Use proper measurement techniques, including maintaining sufficient water movement across the probe face (typically 1 ft. per second)

APPENDIX F – PH MAINTENANCE TIPS AND DETAILS

Lack of electrolyte or old electrolyte

pH probes may have refillable reference electrode reservoirs or sealed, non-refillable reference electrode reservoirs.

- Non-refillable pH probes will need to be replaced periodically. For instance, the pH probes from YSI have a shelf-life of approximately 18-24 months. In general, YSI probes should be replaced about every 2 years. Consult the manufacturer for diagnostic procedures to check the lifespan of the probe.
- Refillable pH probes should be checked frequently to ensure electrolyte is at a suitable level and that no excessive crystallization is present. Excessive crystallization may prevent proper electrolyte flow. If crystallization is observed in the electrode, clean per the manufacturer's instructions.

Clogged reference junction

The reference junction must be able to allow contact between the sample and the reference solution within the probe. A blocked reference junction will produce erroneous measurements. All personnel using the meter should be aware of the location and condition of the reference junction. A black reference junction often indicates that cleaning is required. Follow the manufacturer's instructions for cleaning or replacing the junction.

Dirty or scratched pH bulb

The glass on the pH bulb is very fragile and must be protected. The glass bulb should be clear. A whitish or oily film is indicative of a dirty bulb. Slow response time often indicates that the glass bulb is dirty. Follow the manufacturer's instructions for cleaning the glass bulb. A scratched bulb often results in jumpy, erratic, or impossible readings. Scratched bulbs generally require replacement of the entire probe.

Improper storage

Follow the manufacturer's instructions for probe storage. Most manufacturers recommend storing the pH probe in pH 4 or pH 7 buffer. Never store a pH probe in distilled water. This allows ions to leach out from inside the probe. To restore a pH probe that had been stored in distilled water, most manufacturers recommend soaking the probe in pH 4 buffer. At least several hours to overnight soaking are typically required for restoration.

pH Meter Troubleshooting

Table 3 lists potential remedies for the following symptoms:

- Unable to calibrate (error message during calibration)
- Jumpy, erratic, or questionable readings
- Slow response time
- Failed calibration check

Table 3: pH Meter Troubleshooting

Potential pH problem	Solution
Lack of electrolyte or expired electrolyte	Refill electrolyte (refillable probes only) or replace probe (non-refillable)
Clogged reference junction	Clean or replace reference junction
Dirty glass bulb	Clean bulb
Scratched glass bulb	Replace probe
pH probe stored dry or in DI water	Soak pH probe in pH 4 buffer or electrolyte solution. Soaking for several hours or overnight is typically needed.
Contaminated standard	Use fresh standard. Rinse probes thoroughly between uses and with a small amount fresh standard before submersing.

APPENDIX G – CONDUCTIVITY MAINTENANCE TIPS AND DETAILS

Conductivity Cell Condition

A clean conductivity cell is one of the most important requirements for obtaining accurate measurements. Rinsing with deionized water after each use is recommended. Follow the manufacturer's instructions for cleaning the conductivity cell. General cleaning instructions may include:

- Rinse the conductivity cell frequently with deionized water.
- Pay diligent attention that no dirt or debris remains inside the conductivity cell.
- Cleaning the cell with a q-tip or soft-bristled brush and isopropyl alcohol or a mild detergent. For more resistant residues, check the manufacturer's instructions.

Zero Check Point

A dry conductivity cell should read 0 in air. High quality distilled water has a conductivity in the range of 0.5 to 3 µS/cm. Performing a check of the conductivity cell in dry air is a good way to quickly check the zero point of the meter. If the meter does not read 0 in air, it should be cleaned thoroughly. If the meter will not read close to 0 in air after extensive cleaning, and will not calibrate or pass the low level check, it may need repair or replacement.

Improper Calibration or Measurement Procedure

For new users, improper calibration or measurement procedures are a common source of error. The two most common mistakes are:

- Not fully submersing the probe: Artificially low readings will result if the probe is not fully submerged.
- Bubbles trapped inside cell: Air bubbles trapped inside the cell may give erroneous readings. The enclosed, tubular design of some conductivity cells often trap air bubbles that the user cannot see. Gently agitate the probe to remove any air bubbles that may be trapped in the cell.

Failed Conductivity Checks

If the meter fails the conductivity check, rinse the cell repeatedly with deionized water, dry, and ensure the probe reads close to zero in air. Then repeat the check in

fresh standard. A few reasons why a check might fail are: 1.) an initial bad calibration; 2.) contaminated standard; 3.) improper technique; or 4.) dirty conductivity cell.

Conductivity Meter Troubleshooting

Table 4 lists some potential remedies for the following symptoms:

- Unable to calibrate (error message during calibration)
- Questionable readings
- Conductivity unstable or inaccurate
- Failed conductivity check

Table 4: Conductivity Meter Troubleshooting

Potential problem	Solution
Dirty conductivity cell	Clean cell per manufacturer's instructions Confirm probe reads close to zero in dry air
Contaminated standards	Clean cell per manufacturer's instructions Use fresh, certified standards Never reuse standards Low level-standards are easily contaminated; use with extra caution Check expiration date of standard Rinse probes thoroughly between uses and with a small amount fresh standard before submersing in the standard
Improper calibration technique cell not fully submerged bubbles in conductivity cell	Follow manufacturer's instructions carefully
Temperature-compensation error	Ensure automatic temperature compensation (ATC) factor programmed in meter matches calibration solution Follow ATC check for conductivity in DWR Wastewater/Groundwater Laboratory Certification Guidelines Return meter to manufacturer for repair if it fails ATC check

APPENDIX H – PRESERVATION AND HOLD TIME TABLE

Surface Water Protection Preservation and Hold Time Table

Aqueous Samples				
Listed below is information on the collection and preservation of samples. The amount of sample listed is for routine conditions. If you suspect that unusual conditions or interferences exist, please submit double the amount of sample. Excluding purgeable organics and sulfide, a one-half inch air space should be left in all bottles to allow for mixing before analysis. The parameters are listed in the same order as they appear on the DM-1 form.				
Parameter [1]	**Minimum Required Volume**	**Container** [13]	**Preservation** [21]	**Maximum Hold Time** [22]
BOD, 5-day	1 liter	P	Cool, ≤6°C	48 hours [2]
CBOD, 5-day	1 liter	P	Cool, ≤6°C	48 hours [2]
COD	200 mL	P (Disposable)	Cool, ≤6°C, 25% H_2SO_4 to pH<2	28 days
Coliform (Total, Fecal, *E. coli* and Enterococci)	250 mL **each**	P (Sterile) (3)	Cool, <10°C, 0.008% $Na_2S_2O_3$ (0.1 mL 10% $Na_2S_2O_3$ per 125 mL) and 15% EDTA [3]	6 hours [4]
Residue (TS, TSS) [18]	500 mL **each**	P (Disposable)	Cool, ≤6°C	7 days
Total Dissolved Solids (TDS)	500 mL each	P (Disposable)	Cool, ≤6°C	7 days
pH [5]	Inappropriate for laboratory analysis			Immediate - field measurement
A. Acidity [18]	200 mL	P (Disposable)	Cool, ≤6°C	14 days
A. Alkalinity [18]	200 mL	P (Disposable)	Cool, ≤6°C	14 days
A. Bicarbonate	combined w/above	Request on Field Sheet and submit alkalinity sample.		
A. Carbonate	combined w/above	Request on Field Sheet and submit alkalinity sample.		
TOC	200 mL	P (Disposable)	Cool, ≤6°C, H_3PO_4 to pH<2	28 days
DOC	200 mL - A Field Blank must accompany all DOC samples [26]	P (Disposable)	Field filter using 0.45 µm filter, Cool, ≤6°C, H_3PO_4 to pH<2	28 days
Turbidity	200 mL	P (Disposable)	Cool, ≤6°C	48 hours [2]
C. Chloride	500 mL x 1	P (Disposable)	Cool, ≤6°C when combined with SO_4 - no thermal preservation required if requesting chloride only	28 days
C. Fluoride	combined w/above	P (Disposable)	Cool, ≤6°C when combined with SO_4 - no thermal preservation required if requesting fluoride only	28 days
C. Sulfate	combined w/above	P (Disposable)	Cool, ≤6°C	28 days
Chlorophyll a [10]	500 mL	P (Brown, wide-mouth bottle)	Cool, ≤6°C [12] [24]	24 hours [12] [24]
Color	200 mL	P (Disposable)	Cool, ≤6°C	48 hours [2]
Chromium, Hexavalent	200 mL	P (Disposable)	Cool, ≤6°C	24 hours (notify lab of collection)
Cyanide, Total	2 liters (two 1-liter bottles)	P	Cool, ≤6°C, 0.6 g ascorbic acid [6], 6N NaOH to pH>12	14 days [19]
Formaldehyde	500 mL	P (Disposable)	Cool, ≤6°C	N/A
HEM: Oil and Grease	2 liters (two 1-liter bottles) [17]	G (Wide-mouth quart jar, Teflon-lined cap)	Cool, ≤6°C, 6N H_2SO_4 to pH<2	28 days
Total Hardness (request by checking Ca and Mg on field sheet - can be part of metals suite) **Total Hardness (mg $CaCO_3$/L) = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]**	500 mL	P (Disposable)	1+1 HNO_3 to pH<2	6 months
MBAS	500 mL	P (Disposable)	Cool, ≤6°C	48 hours [2] (notify lab of collection)

Surface Water Protection Preservation and Hold Time Table

Parameter [1]	Minimum Required Volume	Container [13]	Preservation [21]	Maximum Hold Time [22]
Phenols, Total Recoverable	2 liters (two 1-liter bottles)	G (Phenol bottle) only	Cool, ≤6°C, 1:1 H2SO4 to pH<2 (1 mL ferrous ammonium sulfate if sample contains oxidizer)	28 days
Sulfide	40 mL x 3 [9]	G (40 mL VOA vials with Teflon-lined septum)	Cool, ≤6°C, add 0.1 mL 2N zinc acetate plus 6N NaOH to pH>9, leave NO headspace in the bottle	7 days
Specific Conductance	200 mL	P (Disposable)	Cool, ≤6°C	28 days
Tannin and Lignin	500 mL	P (Disposable)	Cool, ≤6°C	28 days
B. NH3 as N	500 mL x 1	P (Disposable)	Cool, ≤6°C, 25% H2SO4 to pH<2 [7] (0.008% Na2S2O3 if chlorine present) [11]	28 days
B. TKN as N	combined w/above	P (Disposable)	Cool, ≤6°C, 25% H2SO4 to pH<2 [7] (0.008% Na2S2O3 if chlorine present) [11]	28 days
B. NO3+NO2 as N	combined w/above (except when NH3 and TKN require dechlorination)	P (Disposable)	Cool, ≤6°C, 25% H2SO4 to pH<2 [7]	28 days
B. TP, total as P	combined w/above (except when NH3 and TKN require dechlorination)	P (Disposable)	Cool, ≤6°C, 25% H2SO4 to pH<2 [7]	28 days
TP, dissolved as P	200 mL	P (Disposable)	Filter immediately, Cool, ≤6°C, 25% H2SO4 to pH<2 [7]	28 days
PO4 as P	200 mL	P (Disposable)	Filter immediately, Cool, ≤6°C	48 hours [2]
NO2 as N	200 mL	P (Disposable)	Cool, ≤6°C	48 hours (notify lab of collection)
NO3 as N	200 mL + additional preserved sample for NO3+NO2	P (Disposable)	Cool, ≤6°C	48 hours (notify lab of collection)
C. Metals: Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr (Total), Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Sb, Sn, Se, Tl, V, Zn and Hg (20)	500 mL x 1	P (Disposable)	1+1 HNO3 to pH<2 [27]	6 months (28 days for Mercury)
Boron [25]	500 mL x 1	P (Disposable)	1+1 HNO3 to pH<2	6 months
EPA 1631 E Hg (trace level total Hg)	500 mL - A Field Blank must accompany each trace-level Hg sample	G (Borosilicate with Teflon-lined cap)	None required - Use "clean" sampling techniques as described in EPA Method 1669	28 days until preservation with BrCl [23] Preserved samples are stable for up to 90 days from collection
Semivolatile Organics - Base/Neutral Acid Extractables	1 gal	G (Amber with Teflon-lined cap)	Cool, ≤6°C, 0.008% Na2S2O3 if chlorine present) [11]	7 days until extraction [8] 40 days after extraction
Pesticides/PCBs (OP Pest/OC Pest/ON Pest)	1 gal	G (Amber with Teflon-lined cap)	Cool, ≤6°C, 0.008% Na2S2O3 if chlorine present) [11]	7 days until extraction [8] [15] 40 days after extraction

Surface Water Protection Preservation and Hold Time Table

Parameter [1]	Minimum Required Volume	Container [13]	Preservation [21]	Maximum Hold Time [22]
Acid Herbicides	1 gal	G (Amber with Teflon-lined cap)	Cool, ≤6°C, 0.008% Na2S2O3 if chlorine present [11]	7 days until extraction [8] [15] 40 days after extraction
Purgeable (Volatile) Organics (VOA)	40 mL x 4 [9] - A Trip Blank (3 vials) must accompany all VOA samples	G (Teflon-lined septum)	Cool, ≤6°C, 0.008% Na2S2O3 if chlorine present) [11], HCl to pH<2 [14] [16], Leave no headspace in the bottle	14 days (7 days for aromatics only when unpreserved)
TPH Gasoline Range Organics and BTEX (aqueous)	40 mL x 4 [9] - A Trip Blank (3 vials) must accompany all VOA samples	G (Teflon-lined septum)	Cool, ≤6°C, 0.008% Na2S2O3 if chlorine present) [11], HCl to pH<2 [14] [16], Leave no headspace in the bottle	14 days
TPH Diesel Range Organics (aqueous)	1 gal	G (Teflon-lined cap)	Cool, ≤6°C	14 days until extraction 40 days after extraction

Footnotes
(1) Determinations preceded by the same letter (i.e., A, B, C) may be submitted in the same bottle if the bottle contains enough sample. If not letter precedes a parameter, it must be submitted in a separate bottle .
(2) 48 hours is the maximum holding time, however, samples should be submitted to the lab as soon as possible.
(3) Use the 250 mL wide-mouth sterile plastic bottles for all samples. All bottles contain soium thiosulfate and EDTA reagents.
(4) Litigation samples must be delivered to the laboratory within 6 hours of sample collection.
(5) It is recommended that pH analysis be performed on-site.
(6) Add 0.6 g ascorbic acid only if the sample ccontains total residual chlorine.
(7) Caution: Addition of excessive amounts of acid will interfere with the test procedures. The 2.0 mL of 25% H2SO4 per 500 mL sample should be added using a graduated or precise volume dispensing device. If no dispenser is avilable you may add exactly 40 drops of the 25% H2SO4. In most cases, the addition of 2.0 ml (~40 drops) of 25% H2SO4 to 500 mL of surface water will reduce the pH to <2, however, if the pH remains above 2, add acid dropwise with stirring until the pH is lowered to <2. For nutrient samples, the pH range of 1.5-2.0 is ideal to insure best possible recovery of analytes.
(8) In a glass container, submit a small quantity of the pure compound of any suspected material.
(9) Fill the bottle to overflowing and cap, leaving no air space (i.e., headspace).
(10) EPA Method 445.0, Revision 1.2, September 1997 and EPA Method 446.0, Revision 1.2, September 1997.
(11) Should only be used in the presence of residual chlorine. Add sodium thiosulfate or ascorbic acid (as appropriate) to the container first; fill at least half way before adding acid (if used). Adding 0.1 mL of a 10% solution of sodium thiosulfate (Na2S2O3) per each 125 mL of sample is equivalent to 0.008% Na2S2O3.
(12) Used by the DWQ Chemistry Lab only at this time.
(13) The container types listed are those commonly throughout the Department. Other container types may be acceptable. Please consult the laboratory about use of proper containers before deviating from those listed. P-plastic, G-glass, P (Disposable)-plastic disposable "juice" bottle.
(14) Samples submitted for purgeable halocarbons only should not be acid-preserved.
(15) Samples submitted for pesticide and acid herbicide analyses must be extracted within 72 hours of collection if the pH is not adjusted in the lab to a pH range of 5-9.
(16) Samples submitted for purgeable aromatics receiving no pH adjustment must be analyzed witin 7 days of collection.
(17) The entire contents (i.e., whole volume sample) must be used for analysis.
(18) Total Residue and Total Suspended Residue samples are to be shipped directly to the Central Laboratory for repacking and shipment to the Washington Regional Laboratory for analysis. Samples for these parameters collected in the Washington Region are sent directly to the WARO Lab.

Surface Water Protection Preservation and Hold Time Table

(19) Maximum hold time is 24 hours when sulfide is present. Optionally, all samples may be tested on-site with lead acetate paper before pH adjustment in order to determine if sulfide is present. If it is, it can be removed by the addition of CdNO3 powder until a negative spot test is obtained.
(20) For dissolved metals, samples should be filtered immediately on-site before adding preservative.
(21) Sample preservation should be performed immediately upon collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then the samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(22) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Collection times must allow for sample preparation and analytical set-up. Some samples may not be stable for the maximum time period given in the table. Collectors are obligated to hold the sample for as short a time as possible especially if knowledge exists showing that this is necessary to maintain sample stability.
(23) If samples are oxidized (digested) with bromine chloride (BrCl) in the same bottle that they are collected, then the preservation of the sample may be delayed up to twenty-eight days after the time of sample collection. Thte total hold time with proper preservation for EPA Method 1631 is ninety days after collection. Ref: EPA Method 1631, Revision E, Section 8.5.
(24) Samples are cooled to 4°C at the time of collection. Due to the limitations of filtering samples in the field, it is the DWQ Laboratory Section's policy to filter chlorophyll samples the day that the samlpes are received at the lab, not to exceed 24 hours from collection. Filters can be stored frozen in the dark for as long as 3 and 1/2 weeks without significant loss of chlorophyll a.
(25) You must write in "Boron" in one of the blank cells on the field sheet to request Boron analysis and submit a separate metals sample.
(26) A field filter blank should accompany all DOC samples. Samplers may obtain water for this purpose from the Central Laboratory just prior to each sampling event. On the field sheet, write "DIS" next to TOC to request DOC analysis. Complete a separate field sheet for the DOC filter blank.
(27) An aqueous metals sample may be collected and shipped without acid preservation, however, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid
January, 2010

NOTE: Applicable parameters are superseded in this table by Table II of the MUR, published May 18, 2012.

APPENDIX I – COALITION FIELD MONITORING CHECKLIST

COALITION FIELD VISIT CHECKLIST

Date: _____

Coalition Information	DWR Information
Coalition Name:	Name/Position:
_____	_____
Laboratory Name:	Name/Position:
_____	_____
Monitor Name:	Purpose of visit:
_____	_____

METER MAINTENANCE & CALIBRATION

Primary Meter Information (pH, DO, Temp, Conductivity)

Parameter(s): _____

Model/Serial #: _____

Parameter(s): _____

Model/Serial #: _____

- ☐ Meter(s) in working order (reasonably clean and undamaged)
- ☐ Meter(s) stored in clean solution, controlled environment
- ☐ Back-up meter available

Meter Operation & Maintenance

- ☐ Routine maintenance schedule (performed/documented)
- ☐ Maintenance log w/ all meters included
- ☐ Response to meter failure (backup available, spare parts, etc.)

  _____
- ☐ How are anomalous readings identified in the field? What is the response?

  _____

Calibration

Pre-sampling

- ☐ Standards/buffers in preferred values, sufficient volume, w/in expiration
- ☐ Performed in stable environment (temperature, etc.)
- ☐ Meters and probes checked (DO membrane intact, pH bulb clear, conductivity cell clean)
- ☐ Sufficient time allowed for DO probe stabilization
- ☐ Conductivity calibration value: _____ check value: _____
- ☐ Conductivity check w/in 10% of standard
- ☐ Two-point pH calibration (7 and ____)
- ☐ pH check w/in 0.1 SU of buffer

Post-sampling Check

- ☐ Performed in stable environment at end of sampling day
- ☐ Conductivity check w/ two standards - Check value: _____ Check value: _____
- ☐ Conductivity check w/in 10% of standards
- ☐ pH check with two buffers (7 and ____)
- ☐ pH check w/in 0.1 SU of buffers
- ☐ DO check w/in 0.5 mg/L of theoretical value

Calibration Sheet

- ☐ All required information recorded
- ☐ Values checked w/in parameter QC ranges

Comments & Questions:

_____

_____

_____

SAMPLING METHODS

Station Location

- ☐ Stations in documented locations (record lat/long and comments on Station sheet)

Sampling Supplies & Equipment

- ☐ Stored in clean environment
- ☐ Equipment in clean and working condition
- ☐ Proper equipment available

General Sampling Methods

- ☐ Site conditions visible before/during sampling
- ☐ Monitor takes notes regarding environmental and site conditions (precipitation, weather, trash, condition of water, etc.)
- ☐ Sample containers stored in a clean environment
- ☐ Precautions taken to avoid contamination of sample containers

Sample Preservation

- ☐ Samples preserved w/in 15 min of collection (fecal, nutrients & metals)
- ☐ Temperature blank in each cooler

Field Measurements

- ☐ Field measurements conducted in situ
- ☐ Sampling conducted from the upstream side of bridge
- ☐ Sampling conducted in main stream flow from bank
- ☐ Field conditions and measurements documented on site

Sample Collection

TSS/Turbidity

- ☐ Grab sample

Fecal

- ☐ Grab sample in sterile container
- ☐ Sodium thiosulfate for preservation

Nutrients

- ☐ Grab sample
- ☐ 25% H_2SO_4 to pH <2 for preservation

QC Samples

- ☐ QC samples collected (other than Temp blank)_____

Sample Transport & Submittal

- ☐ Coolers filled w/ ice
- ☐ Samples put in coolers w/in 15 min of collection
- ☐ Bottles packed appropriately to minimize chance of breakage and contamination
- ☐ Courier pick-up or monitor drop-off to laboratory under COC procedures
- ☐ Transported in time to meet holding times (8 hrs fecal, 24 hrs chlorophyll, 48 hrs turbidity)

Comments & Questions:

_____

_____

_____

SAFETY

- ☐ Blinkers
- ☐ Flashing beacon/strobe
- ☐ Orange vest
- ☐ First Aid kit
- ☐ Fire extinguisher
- ☐ Safety cones (if necessary)
- ☐ Gloves (available if needed)
- ☐ Acid ampule disposal container
- ☐ Safety glasses
- ☐ Portable eyewash or water

NOTES TO REVIEW W/ MONITOR:

_____